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UNITED STATES DEPARTMENT OF AGRICULTURE

BULLETIN No. 314

Contribution from Office of Public Roads and Rural Engineering
LOGAN WALLER PAGE, Director

Washington, D. C. PROFESSIONAL PAPER December 10, 1915

METHODS FOR THE EXAMINATION OF BITUMINOUS ROAD MATERIALS

By

PRÉVOST HUBBARD, Chemical Engineer, and
CHARLES S. REEVE, Chemist

CONTENTS

	Page
Classification of Materials	2
Scheme of Examination	2
Specific Gravity Determination	4
Specific Viscosity Determination	7
Float Test	9
Penetration Test	11
Melting Point Determination	14
Flash and Burning Points	16
Volatilization Test	19
Distillation Test	21
Dimethyl Sulphate Test	25
Bitumen Soluble in Carbon Disulphide	25
Bitumen Insoluble in Paraffin Naphtha	28
Bitumen Insoluble in Carbon Tetrachloride	30
Fixed Carbon	30
Paraffin Scale	32
Extraction of Bituminous Aggregates	35
Grading the Mineral Aggregate	38
Voids in the Mineral Aggregate	39
Bituminous Emulsions	41
Appendix	43

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CONTENTS.

	Page.		Page.
Classification of materials	2	Bitumen soluble in carbon disulphide	25
Scheme of examination	2	Bitumen insoluble in paraffin naphtha	28
Specific gravity determination	4	Bitumen insoluble in carbon tetrachloride	30
Specific viscosity determination	7	Fixed carbon	30
Float test	9	Paraffin scale	32
Penetration test	11	Extraction of bituminous aggregates	35
Melting point determination	14	Grading the mineral aggregate	38
Flash and burning points	16	Voids in the mineral aggregate	39
Volatilization test	19	Bituminous emulsions	41
Distillation test	21	Appendix	43
Dimethyl sulphate test	25		

INTRODUCTION.

This bulletin is the first revision of Office of Public Roads Bulletin No. 38, which was issued July 27, 1911. Its object is to present a description of methods now in use by the Office of Public Roads and Rural Engineering for the examination of bituminous road materials in such form that, with a little practice and proper equipment, such examinations may be made by any intelligent person. The various tests have, therefore, been described rather more in detail than would be necessary if they were intended for the use of chemists only, and illustrations of practically all of the apparatus required have also been included.

Since the publication of Bulletin No. 38 considerable progress has been made in the standardization of methods of examining bituminous road materials. Many of the methods described in Bulletin No. 38 have been generally adopted. Certain of these methods have, however, been improved during the past four years and the constant demand for this bulletin has led to its present revision. The changes and additions noted below are the result of investigations conducted in the laboratories of the office and of cooperative work with certain technical societies.

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Special attention is called to modifications in the penetration test, determination of fixed carbon, and determination of paraffin scale; and to the substitution of new methods for the old distillation tests and for determination of voids in the mineral aggregate. In addition descriptions of the following methods, which were not included in Bulletin No. 38, are given:

(1) Determination of flash and burning points—open cup method.
(2) Dimethyl sulphate test.
(3) Methods of examining bituminous emulsions.

While it is realized that the following scheme of examination is not perfect and may in the future be improved, it has nevertheless been of great assistance in classifying bituminous road materials and determining their suitability for use according to various methods of application and construction.

CLASSIFICATION OF BITUMINOUS ROAD MATERIALS.

For the purpose of examination bituminous road materials may be classified under the following headings:

1. Petroleums and petroleum products, including heavy distillates, malthas, residual petroleums, fluxes, oil-asphalts, and fluxed or cut-back oil-asphalts.
2. Asphalts and other solid native bitumens, and asphaltic cements produced by fluxing them.
3. Petroleum and asphalt emulsions.
4. Tars and tar products.
5. Mixtures of tar with petroleum or asphalt products.
6. Bituminous aggregates, including rock asphalts or bituminous rocks, bituminous concrete, asphalt block, and bituminous topping.

SCHEME OF EXAMINATION.

All petroleum, maltha, and solid native bitumen products are subjected to the following tests:

Specific gravity.
Volatilization at 163° C.
Bitumen soluble in carbon disulphide.
Bitumen insoluble in 86° B. paraffin naphtha.
Fixed carbon.

Of these types the very fluid and sometimes the more viscous products may be subjected to the viscosity, flash, and burning-point determinations. Very viscous materials, too soft for the penetration test, are subjected to the float test, and semisolid and solid products to the penetration test. If the material is sufficiently hard at ordinary temperatures, a melting-point determination may also prove of value. Sometimes two or more of the above-mentioned tests, depending upon the character of the material and the use to which it is to be put, may be made to advantage on a single material. When for any reason it is suspected that the material under examination has been overheated

and possibly injured during process of manufacture, or prepared from a solid native indurated bitumen, the determination of bitumen insoluble in carbon tetrachloride may be made. The paraffin scale determination is made on those materials which are to be identified as being partly composed of heavy paraffin hydrocarbons. The residue obtained from the volatilization test is usually subjected to either the float or penetration test, and in addition it may be subjected to any or all of the above-described tests as occasion may require.

Tar and tar products are subjected to the following tests:

Specific gravity.
Distillation.
Bitumen soluble in carbon disulphide.

Petroleum and asphalt emulsions are subjected to some of the methods of examination applicable to fluid and viscous residual petroleums and also to the following tests:

Determination of water.
Determination of ammonia.
Determination of fixed alkali.
Determination of fatty and resin acids.

In addition, the viscosity test may be employed for fluid products and it is highly desirable that the float test be made on all of the viscous and semisolid tar products. The more or less solid refined tars or tar pitches are also subjected to the melting-point determination. Mixtures of tar and petroleum or asphalt products are in addition subjected to the dimethyl sulphate test.

Some exceptional materials can not be satisfactorily examined according to any one predetermined scheme, and at the present time this matter must be left to the judgment and experience of the analyst. Practically all of the methods described in this bulletin are, however, applicable to the more common materials, and for a given material those methods should be selected which will give the most information concerning its character and suitability for the specific use for which it is intended.

Bituminous aggregates are first of all examined for the percentage of bitumen soluble in carbon disulphide. If the amount is in excess of 5 per cent, an extraction is then made on a large sample and the recovered bitumen is examined according to one of the above-mentioned schemes if it can be identified, or, if not, it is subjected to those tests which are of most value as suggested above. The extracted mineral aggregate is usually quantitatively graded and, if it is to be used or has been used as an integral part of the road proper, its percentage of voids is sometimes determined.

Forms for reporting the results of examination of bituminous road materials according to the methods described in this bulletin are given in the appendix.

SPECIFIC GRAVITY DETERMINATION.

HYDROMETER METHOD USED FOR THIN FLUID BITUMENS.

EQUIPMENT.

1 hydrometer jar approximately 35 centimeters long and 5 centimeters in diameter. (Fig. 1–*a*.)
1 1-pint tin cup, seamless type. (Fig. 1–*b*.)
1 enamel-ware dish approximately 2 inches deep and 9 inches in diameter. (Fig. 1–*c*.)
1 chemical thermometer reading from —10° C. to 110° C. (Fig. 1–*d*.)
1 set of hydrometers—those with a double scale at 15.5° C. (60° F.), one for Baumé and one for a direct specific gravity reading to the third decimal place are used by the Office of Public Roads and Rural Engineering. (Fig. 1–*e*.)
1 hydrometer reading from 0.800 to 0.900 specific gravity.
1 hydrometer reading from 0.900 to 1.000 specific gravity.
1 hydrometer reading from 1.000 to 1.200 specific gravity.
1 hydrometer reading from 1.200 to 1.400 specific gravity.

METHOD.

The specific gravity of thin fluid bituminous road materials is determined at 25° C. as compared with water at that temperature.

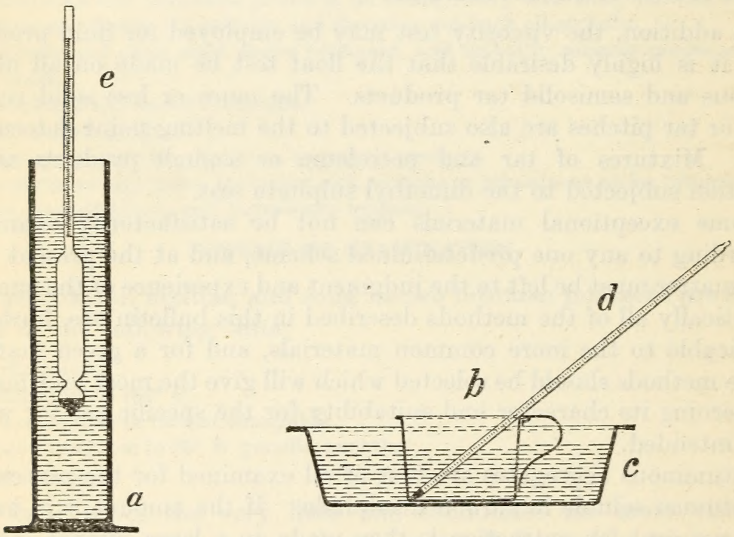


FIG. 1.—Hydrometer method of determining specific gravity.

This may be done with the above-mentioned apparatus by first pouring a sufficient quantity of the material into the tin cup, which is then placed in the large dish containing cold or warm water as occasion may require. The material in the cup should be stirred with the thermometer until it is brought to a temperature of 25° C., after which it should be immediately poured into the hydrometer jar and its gravity determined by means of the proper hydrometer. In case

the hydrometer sinks slowly, owing to the viscosity of the material, it should be given sufficient time to come to a definite resting point, and this point should be checked by raising the hydrometer and allowing it to sink a second time. The hydrometer should never be pushed below the point at which it naturally comes to rest until the last reading has been made. It may then be pushed below the reading for a distance of three or four of the small divisions on the scale, whereupon it should immediately begin to rise. If it fails to do so, the material is too viscous for the hydrometer method, and the pycnometer method should be employed.

The direct specific gravity reading obtained by the foregoing method is based upon water at 15.5° C. taken as unity. For all practical purposes this reading may be corrected to water at 25° C., considered as unity, by multiplying it by 1.002. Thus:

Specific gravity 25° C./25° C. = specific gravity 25° C./15.5° C. × 1.002

PYCNOMETER METHOD (USED FOR VISCOUS FLUID AND SEMISOLID BITUMENS AND EMULSIONS).

EQUIPMENT.

1 large metal kitchen spoon.
1 steel spatula or kitchen knife.
1 Bunsen burner and rubber tubing.
1 250 cubic centimeter low-form glass beaker.
1 chemical thermometer reading from −10° C. to 110° C.
1 special pycnometer. (Fig. 2.)
1 analytical balance, capacity 100 grams, sensitive to 0.1 milligram.

METHOD.

The inconvenience and difficulty of employing the ordinary narrow-neck pycnometer when determining the specific gravity of viscous fluid and semisolid bitumens has led to the use of the special form shown in figure 2.

This pycnometer consists of a fairly heavy, straight-walled glass tube, 70 millimeters long and 22 millimeters in diameter, carefully ground to receive an accurately fitting solid glass stopper with a hole of 1.6 millimeters bore in place of the usual capillary opening. The lower part of this stopper is made concave in order to allow all air bubbles to escape through the bore. The depth of the cup-shaped depression is 4.8 millimeters at the center. The stoppered tube has a capacity of about 24 cubic centimeters and when empty weighs about 28 grams. Its principal advantages are (1) that any desired amount of bitumen may be poured in without touching the sides above the level desired; (2) it is easily cleaned; (3) on account of the 1.6-millimeter bore the stopper can be more easily inserted when the tube is filled with a very viscous oil than if it contained a capillary opening.

When working with semisolid bitumens which are too soft to be broken and handled in fragments, the following method of determining their specific gravity is employed. The clean, dry pycnometer is first weighed empty and this weight is called "a." It is then filled in the usual manner with freshly distilled water at 25° C., and the weight is again taken and called "b." A small amount of the bitumen should be placed in the spoon and brought to a fluid condition by the gentle application of heat, with care that no loss by evaporation occurs. When sufficiently fluid, enough is poured into the dry pycnometer, which may also be warmed, to fill it about half full, without allowing the material to touch the sides of the tube above the desired level. The tube and contents are then allowed to cool to room temperature, after which the tube is carefully weighed with the stopper. This weight is called "c." Distilled water, at 25° C., is then poured in until the pycnometer is full. After this the stopper is inserted, and the whole cooled to 25° C. by a 30-minute immersion in a beaker of distilled water maintained at this temperature. All surplus moisture is then removed with a soft cloth, and the pycnometer and contents are weighed. This weight is called "d." From the weights obtained the specific gravity of the bitumen may be readily calculated by the following formula:

FIG. 2.—Pycnometer (Hubbard type).

$$\text{Specific gravity } 25° \text{ C.}/25° \text{ C.} = \frac{c-a}{(b-a)-(d-c)}$$

Both "a" and "b" are constants and need be determined but once. It is therefore necessary to make but two weighings for each determination after the first. Results obtained according to the method given above are accurate to within 2 units in the third decimal place, while the open-tube method is accurate to the second decimal place only.

The specific gravity of fluid bitumens may be determined in the ordinary manner with this pycnometer by completely filling it with the material and dividing the weight of the bitumen thus obtained by that of the same volume of water.

The pycnometer may be readily cleaned by placing it in a hot-air bath until the bitumen is sufficiently fluid to pour. As much is drained out as possible and the interior swabbed with a piece of cotton waste. It is then rinsed clean with a little carbon disulphide, and after drying is again ready for use.

DISPLACEMENT METHOD (USED FOR HARD SOLID BITUMENS).

EQUIPMENT.

1 chemical thermometer reading from −10° C. to 110° C.
1 analytical balance, capacity 100 grams, sensitive to 0.1 milligram.
1 wood or metal platform.
1 150 cubic centimeter low-form glass beaker.
1 piece of fine silk thread.

METHOD.

For materials which are hard enough to be broken and handled in fragments at room temperature, the following method will prove convenient. A small fragment of the bitumen (about 1 cc.) is suspended by means of a silk thread from the hook on one of the pan supports, about 1½ inches above the pan, and weighed. This weight is called "a." It is then weighed immersed in water at 25° C., as shown in figure 3, and this weight is called "b." The specific gravity may then be calculated by means of the following formula:

$$\text{Specific gravity} = \frac{a}{a-b}$$

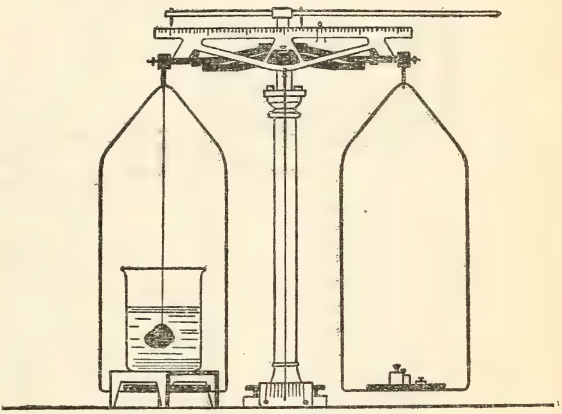


FIG. 3.—Displacement method of determining specific gravity.

USE OF SPECIFIC GRAVITY DETERMINATION.

The specific gravity determination is made on all bitumens containing less than 50 per cent mineral matter, and also on bitumens recovered from bituminous aggregates. The specific gravity is usually reported to the third decimal place.

SPECIFIC VISCOSITY DETERMINATION.

EQUIPMENT.

1 Engler viscosimeter complete with thermometers, burner, and rubber tubing.
1 100 cubic centimeter cylindrical glass graduate.
1 stop watch.

METHOD.

The viscosity of fluid bituminous road materials may be determined at any suitable temperature by means of the Engler viscosimeter. This apparatus is shown in figure 4, and may be described as follows: *a* is a brass vessel for holding the material to be tested, and may be

closed by the cover *b*. To the conical bottom of *a* is fitted a conical outflow tube *c*, exactly 20 millimeters long, with a diameter at the top of 2.9 millimeters and at the bottom of 2.8 millimeters. This tube can be closed and opened by the pointed hardwood stopper *d*. Pointed metal projections are placed on the inside of *a* at equal distances from the bottom and serve for measuring the charge of material, which is 240 cubic centimeters. The thermometer *e* is used to ascertain the temperature of the material to be tested. The vessel *a* is surrounded by a brass jacket *f*, which holds the material used as a heating bath, either water or cottonseed oil, according to the temperature at which the test is to be made. A tripod *g* serves as a support for the apparatus and also carries a ring burner *h* by means of which the bath is directly heated. The measuring cylinder of 100 cubic centimeters capacity, which is sufficiently accurate for work with road materials, is placed directly under the outflow tube.

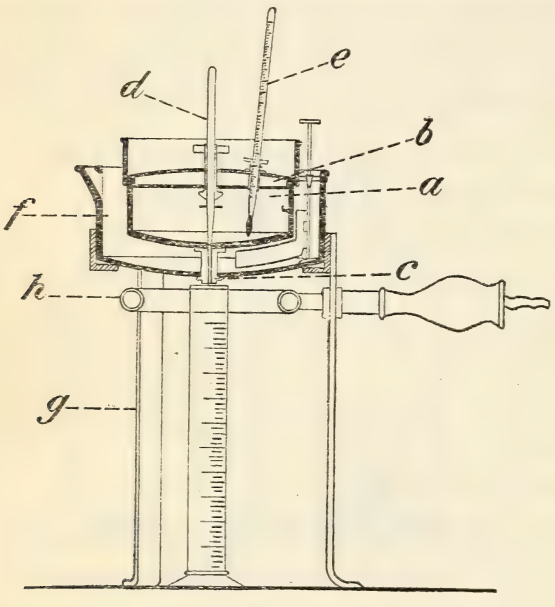


FIG. 4.—Engler viscosimeter.

As all viscosity determinations should be compared with that of water at 25° C., the apparatus should be previously calibrated as follows: The cup and outlet tube should first be scrupulously cleaned. A piece of soft tissue paper is convenient for cleaning the latter. The stopper is then inserted in the tube and the cup filled with water at 25° C. to the top of the projections. The measuring cylinder should be placed directly under the outflow tube so that the material, upon flowing out, will not touch the sides, and the stopper may then be removed. The time required both for 50 and 100 cubic centimeters to run out should be ascertained by means of a stop watch and the results so obtained should be checked a number of times. The time required for 50 cubic centimeters of water should be about 11 seconds and for 100 cubic centimeters about 22.8 seconds.

Bituminous road materials are tested in the same manner as water and the temperature at which the test is made is controlled by the bath. The material should be brought to the desired temperature

and maintained there for at least three minutes before making the test. The results are expressed as specific viscosity compared with water at 25° C., as follows:

$$\text{Specific viscosity at } a^\circ \text{ C.} = \frac{\text{seconds for passage of given volume at } a^\circ \text{ C.}}{\text{seconds for passage of same volume of water at } 25^\circ \text{ C.}}$$

USE OF VISCOSITY DETERMINATION.

For all thin fluid bituminous road materials the specific viscosity is determined at 25° C. with 50 or 100 cubic centimeters. Viscous fluid products are run at 40° C. or 50° C. with 50 cubic centimeters and very viscous products at 100° C. or over with 50 cubic centimeters. This test is not always made on the materials above mentioned, but is a useful one when they are required to have a given degree of fluidity at a given temperature.

FLOAT TEST.

EQUIPMENT.

1 aluminum float or saucer. (Fig. 5-*a*.)
2 conical brass collars. (Fig. 5-*b*.)
2 1-quart tin cups, seamless.
2 chemical thermometers reading from — 10° C. to 110° C.
1 iron tripod.
1 Bunsen burner and rubber tubing.
1 burette clamp and support.
1 large metal kitchen spoon.
1 steel spatula or kitchen knife.
1 brass plate 5 by 8 centimeters.
1 stop watch.

METHOD.

The float apparatus consists of two parts, an aluminum float or saucer (fig. 5–*a*) and a conical brass collar (fig. 5–*b*). The two parts are made separately, so that one float may be used with a number of brass collars.

In making the test the brass collar is placed with the small end down on the brass plate, which has been previously amalgamated with mercury by first rubbing it with a dilute solution of mercuric chloride or nitrate and then with mercury. A small quantity of the material to be tested is heated in the metal spoon until quite fluid, with care that it suffers no appreciable loss by volatilization and that it is kept free from air bubbles. It is then poured into the collar in a thin stream until slightly more than level with the top. The surplus may be removed, after the material has cooled to room temperature, by means of a spatula or steel knife which has been slightly heated. The collar and plate are then placed in one of the tin cups containing ice water maintained at 5° C., and left in

this bath for at least 15 minutes. Meanwhile the other cup is filled about three-fourths full of water and placed on the tripod, and the water is heated to any desired temperature at which the test is to be made. This temperature should be accurately maintained, and should at no time throughout the entire test be allowed to vary more than one-half a degree centigrade from the temperature selected. After the material to be tested has been kept in the ice water for at least 15 minutes, the collar with its contents is removed from the plate and screwed into the aluminum float, which is then immediately floated in the warmed bath. As the plug of bituminous material

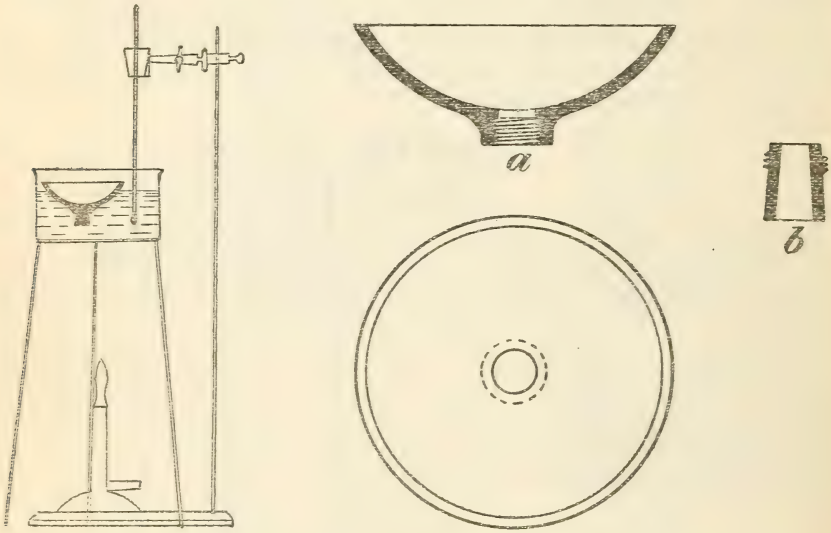


FIG. 5.—New York testing laboratory float apparatus.

becomes warm and fluid, it is gradually forced upward and out of the collar, until water gains entrance to the saucer and causes it to sink.

The time in seconds between placing the apparatus on the water and when the water breaks through the bitumen is determined by means of a stop watch and is taken as a measure of the consistency of the material under examination.

USE OF THE FLOAT TEST.

This test is always made on viscous and semisolid refined tars, and often on the viscous and semisolid petroleum and asphalt products, although, when the penetration test can be employed on the two latter classes of material, the float test is not always considered necessary. For the more fluid products the test is made at 32° C. and for the semisolid materials, at 50° C. When the material under examination is quite hard, the test may be run at 100° C.

The float test is a most convenient one for roughly checking the uniformity of different shipments of bituminous material furnished under specifications.

PENETRATION TEST.

EQUIPMENT.

1 penetrometer complete, with a seconds pendulum or metronome. (Figs. 6 and 7.)
1 tin box approximately 5 centimeters in diameter by 3.5 centimeters in height.
1 large metal kitchen spoon.
1 steel spatula or kitchen knife.
1 glass penetration dish approximately 10 centimeters in diameter by 6 centimeters high.
1 enamel-ware dish approximately 3 inches deep and 9 inches in diameter.
1 chemical thermometer reading from —10° C. to 110° C.

METHOD.

The object of the penetration test is to ascertain the consistency of the material under examination by determining the distance a weighted needle will penetrate into it at a given temperature. A standard needle is employed for this purpose and this needle is usually weighted with 100 grams. The depth of penetration is determined upon the bitumen maintained at 25° C., while the load is applied for five seconds.

The standard needle is made from round, polished, annealed-steel drill rod having a diameter of from 0.0405 to 0.0410 inches. The rod is tapered to a sharp point at one end, with the taper extending back one-fourth inch. It is then highly polished, tempered, and again polished with jewelers' rouge. The finished needle is from $1\frac{3}{4}$ to 2 inches in length and exactly 0.040 inch in diameter. This needle, as made in the laboratory of the office, gives the same results as the old standard No. 2 cambric needle, and possesses the advantage that it can be exactly duplicated and accurately described.

The penetration apparatus shown in figure 6 consists of a standard needle *a*, inserted in a short brass rod, which is held in the aluminum rod *b* by a binding screw. The aluminum rod is secured in a framework so weighted and balanced that, when it is supported on the point of the needle, the framework and rod will stand in an upright position, allowing the needle to penetrate perpendicularly without the aid of a support.

The frame, aluminum rod, and needle weigh 100 grams with the weight *c* on the bottom of the frame, while without the weight they weigh 50 grams. Figure 6 shows the needle and weighted frame, together with side and front views of the entire apparatus, put together and ready for making a penetration. The shelf for the sample is marked *d; e* is the clamp to hold the aluminum rod until it is desired to make a test; and *f* is a button which, when pressed,

opens the clamp. By turning this button while the clamp is being held open, it will lock and keep the clamp from closing until unlocked. The device for measuring the distance penetrated by the needle consists of a rack, with a foot *g*. The movement of this rack turns a pinion, to which is attached the hand which indicates on the dial *h* the vertical distance covered by the rack. One division of the dial corresponds to a movement of 0.1 millimeter by the rack. The rack may be raised or lowered by moving the counterweight *i* up or down. The tin box containing the sample to be tested is marked *k;* this is submerged in water contained in the glass cup in order to maintain a constant temperature.

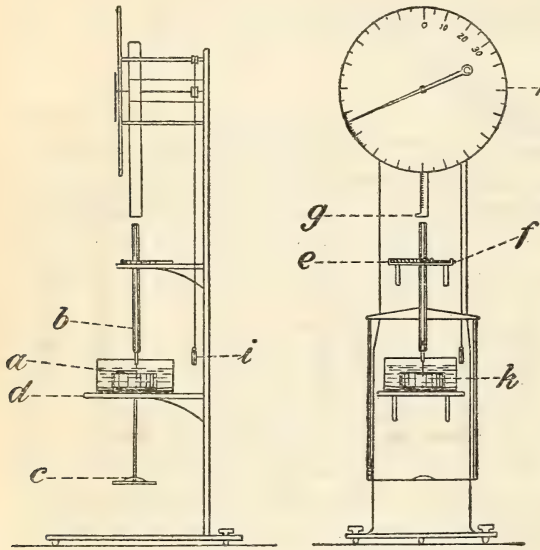


FIG. 6.—Dow penetration machine.

This apparatus is known as the Dow penetration machine. Another type of machine known as the New York Testing Laboratory penetrometer, based upon the same general principle and using the same standards, is at present employed by the Office of Public Roads and Rural Engineering. This penetrometer is shown in figure 7. Both machines give practically the same results, if operated under the same conditions, and it is therefore considered unnecessary to include a description of the latter.

A cup suitable for holding the box containing the test sample during penetration is conveniently made from a glass crystallizing dish 10 centimeters in diameter, with straight sides about 6 centimeters high. Three right triangles with right angle sides 1 and 5 centimeters, respectively, are cut from $\frac{1}{16}$-inch sheet metal. Some solid bitumen is melted in the bottom of the dish forming a layer about $\frac{1}{8}$-inch thick, into which the triangles are placed, resting on the side five centimeters long. Their apexes should meet at the center, with their short sides dividing the circumference of the dish into three equal arcs. When the bitumen has hardened, the triangles give a firm support for circular boxes, and the possibility of any rocking motion and consequent faulty results is avoided.

The penetration test is made as follows: A sample of the material to be tested is first warmed sufficiently to flow, and poured into the tin box. The box and contents, after cooling for one-half hour at room temperature, are immersed in water maintained at the temperature at which the test is to be made, and allowed to remain immersed for one and one-half hours. The sample in the tin box should now be placed in the glass cup and removed in it, covered with as much water as convenient without spilling, to the shelf *d*. The brass rod with the needle is inserted into *b* and secured by tightening the binding screw. The rod is lowered until the point of the needle almost touches the surface of the sample; then by grasping the frame with both hands it is cautiously pulled down until the needle just comes in contact with the surface of the sample. This can be seen best by having a light so situated that, upon looking through the sides of the glass cup, the needle will be reflected from the surface of the sample. After thus setting the needle, the counterweight is slowly raised until the foot of the rack rests on the head of the rod and a reading of the dial taken. The clamp is then opened wide by pressing the button and held in this position for exactly five seconds, as determined by the pendulum or metronome. The clamp is then released, the rack lowered until it rests on the rod, and the difference between the first and second readings of the dial in hundredths of a centimeter is taken as the distance penetrated by the needle.

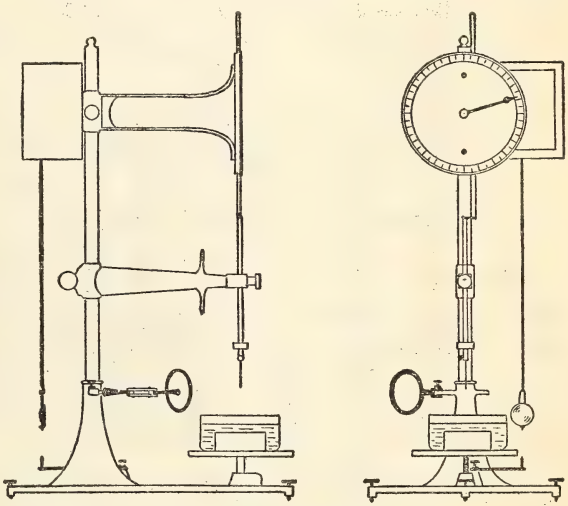


FIG. 7.—New York Testing Laboratory penetrometer.

Owing to the susceptibility of certain bitumens to slight changes in temperature, the water bath should be accurately maintained at the desired temperature, both before and during the test, and, when the room temperature differs greatly from that of the bath, the water in the glass cup should be renewed after each test. An average of from three to five tests, which should not differ more than four points between maximum and minimum, is taken as the penetration of the sample. The tests should be made at points on the surface of the sample not less than one centimeter from the side of the container and not less than one centimeter apart.

The needle should be removed and thoroughly cleaned by wiping with a dry cloth, after which it is ready for another test. The point of the needle should be examined from time to time with a magnifying glass to see that it is not injured in any way. If it is found defective, it may be removed by heating the brass rod and withdrawing with pliers. A new needle may then be inserted in the heated brass rod, and held firmly in place by a drop of soft solder.

USE OF PENETRATION TEST.

This test is made on all semisolid and solid oil-asphalts, asphaltic cements, and native asphalts, but seldom on tar products. It is also often made on the residues of materials subjected to the volatilization tests, when they are sufficiently hard. For work on residues, which seldom amount to more than 20 cubic centimeters, a small container, which should not, however, be less than 1 inch in diameter, will be required.

While the standard conditions under which this test is made call for a 100-gram load applied for five seconds on the material maintained at a temperature of 25° C., it is sometimes desirable, when very soft materials are tested, to make the test with a 50-gram weight. In order to ascertain how susceptible a material may be to temperature changes, tests may be made at any other desired temperatures, preferably 0° C., with a 200-gram weight for one minute, and at 46° C. with a 50-gram weight for five seconds.

In all cases the results of tests should be reported in hundredths of a centimeter, as follows, showing all the conditions in order that no misinterpretation of results may occur:

Penetration (—— seconds, —— grams at ——° C.)= ——.

MELTING POINT DETERMINATION.

EQUIPMENT

1 iron tripod.
1 Bunsen burner and rubber tubing.
1 piece of wire gauze 10 centimeters square.
1 800 cubic centimeter Jena glass beaker, low form. (Fig. 8-*a*.)
1 400 cubic centimeter Jena glass beaker, tall, without lip. (Fig. 8-*b*.)
1 iron ring support (ring 7.5 centimeters in diameter) and burette clamp. (Fig. 8-*c*.)
1 metal cover. (Fig. 8-*d*.)
1 object glass.
1 piece of wire (No. 12 Brown & Sharpe gauge) 20 centimeters in length, bent. (Fig. 8-*e*.)
1 chemical thermometer reading from 0° C. to 250° C.
1 cubical brass mold. (Fig. 8-*f*.)
1 large metal kitchen spoon.
1 steel spatula or kitchen knife.

METHOD.

Since bitumens are mixtures of various organic compounds, they can have no true melting point, but an arbitrary method for determining the so-called melting point of those materials sufficiently solid to maintain their form for some time under normal conditions is of value as a means of identification and for control work. A number of methods have been tried, but the following has been selected as the most convenient and accurate for such materials.

The material under examination is first melted in the spoon by the gentle application of heat until sufficiently fluid to pour readily. Care must be taken that it suffers no appreciable loss by volatilization. It is then poured into the ½-inch brass cubical mold, which has been amalgamated with mercury and which is placed on an amalgamated brass plate. The brass may be amalgamated by washing it first with a dilute solution of mercuric chloride or nitrate, after which the mercury is rubbed into the surface. By this means the bitumen is, to a considerable extent, prevented from sticking to the sides of the mold. The hot material should slightly more than fill the mold and, when cooled, the excess may be cut off with a hot spatula.

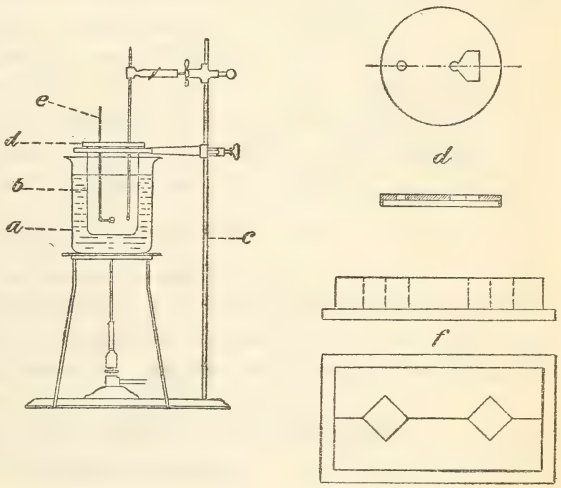


FIG. 8.—Melting point apparatus.

After cooling to room temperature, the mold is placed in a bath maintained at 25° C. for one-half hour. The cube is then removed and fastened upon the lower arm of a No. 12 wire (Brown & Sharpe gauge), bent at right angles and suspended beside a thermometer in a covered Jena glass beaker of 400 cubic centimeters capacity, which is placed in a water bath, or, for high temperatures, a cottonseed-oil bath. The wire should be passed through the center of two opposite faces of the cube, which is suspended with its base 1 inch above the bottom of the beaker. The water or oil bath consists of an 800-cubic centimeter low-form Jena glass beaker suitably mounted for the application of heat from below. The beaker in which the cube is suspended is of the tall-form Jena type without lip. The metal cover has two openings as shown in figure 8–*d*. A cork, through which

passes the upper arm of the wire, is inserted in one hole and the thermometer in the other. The bulb of the thermometer should be just level with the cube and at an equal distance from the side of the beaker. In order that a reading of the thermometer may be made, if necessary, at the point which passes through the cover, the hole is made triangular in shape and covered with an ordinary object glass through which the stem of the thermometer may be seen. Readings made through this glass should be corrected for the angle of observation, which may be made constant by always sighting from the front edge of the opening to any given point on the stem of the thermometer below the cover.

After the test specimen has been placed in the apparatus, the liquid in the outer vessel is heated in such a manner that the thermometer registers an increase of 5° C. per minute. The temperature at which the bitumen touches a piece of paper placed in the bottom of the beaker is taken as the melting point. Determinations made in the manner described should not vary more than 2° for different tests of the same material. At the beginning of this test the temperature of both bitumen and bath should be approximately 25° C.

USE OF MELTING-POINT DETERMINATION.

The melting-point determination should be made on all bituminous road binders sufficiently hard to be handled at room temperature after removing from the mold. This test is not usually required for bitumens which are to be cut with a nonvolatile flux before use.

DETERMINATION OF FLASH AND BURNING POINTS.

CLOSED-CUP METHOD.

EQUIPMENT.

1 New York State Board of Health oil tester with Bunsen burner. (Fig. 9.)
1 open-cup oil tester with Bunsen burner. (Fig. 10.)
1 chemical thermometer reading from 0° C. to 400° C.
1 piece of 6-millimeter glass tubing, 6 centimeters in length, one end of which has been drawn to a 1-millimeter opening. Soft rubber tubing for gas connection.

METHOD.

While for all ordinary purposes the open-cup method of determining the flash and burning points of bituminous road materials is satisfactory, the closed-cup method described below is to be preferred, where greater accuracy is required. This is particularly true for materials of a relatively low flash point.

The oil tester shown in figure 9 consists of a copper oil cup *a* of about 300 cubic centimeters capacity, which is heated in an oil bath *b* by a small Bunsen flame. The cup is provided with a glass cover *c*, carrying a thermometer *d*, and a hole *e* for inserting the

testing flame. The testing flame is obtained from a jet of gas passed through the piece of glass tubing and should be about 5 millimeters in length.

The flash test is made as follows: The oil cup should first be removed and the bath filled with cottonseed oil. The oil cup should be replaced and filled with the material to be tested to within 3 millimeters of the flange joining the cup and the vapor chamber above. The glass cover is then placed on the oil cup and the thermometer so adjusted that its bulb is just covered by the bituminous material. The Bunsen flame should be applied in such a manner that the temperature of the material in the cup is raised at the rate of about 5° C. per minute. From time to time the testing flame is inserted in the opening in the cover to about half way between the surface of the material and the cover. The appearance of a faint bluish flame over the entire surface of the bitumen shows that the flash point has been reached and the temperature at this point is taken.

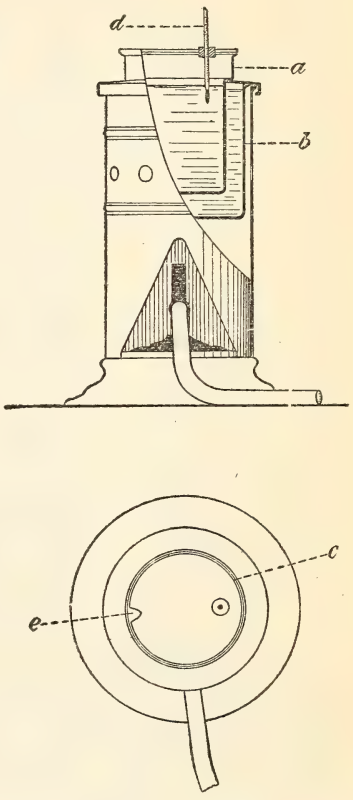


FIG. 9.—New York State Board of Health oil tester.

The burning point of the material may now be obtained by removing the glass cover and replacing the thermometer in a wire frame. The temperature is raised at the same rate and the material tested as before. The temperature at which the material ignites and burns is taken as the burning point.

At the conclusion of this test the flame should not be blown out for danger of splashing the hot material. A metal cover or extinguisher should be employed for this purpose by placing it over the ignited material.

OPEN-CUP METHOD.

A number of open-cup oil testers have been devised which are similar in design and give practically equivalent results. This type is shown in figure 10. It consists of a brass oil cup *a* of about 100 cubic centimeters capacity. The outer vessel *b* serves as an air jacket. No glass cover is used in the open-cup method. A suitable

thermometer *c* is suspended from the wire support *d* directly over the center of the cup so that its bulb is entirely covered with oil but does not touch the bottom of the cup. The testing flame is obtained from a jet of gas passed through a piece of glass tubing, and should be about 5 millimeters in length.

The test is made by first filling the oil cup with the material under examination to within about 5 millimeters of the top. The Bunsen flame is then applied in such a manner that the temperature of the material in the cup is raised at the rate of 5° C. per minute. From time to time the testing flame is brought almost in contact with the surface of the oil. A distinct flicker or flash over the entire surface of the oil shows that the flash point is reached and the temperature at this point is taken. It will usually be found that the flash point as determined by the open-cup method is somewhat higher than by the closed-cup method, for the same material.

The burning point of the material is obtained by continuing the test and noting that temperature at which it ignites and burns. The flame should then be extinguished by means of a metal cover supplied with the instrument.

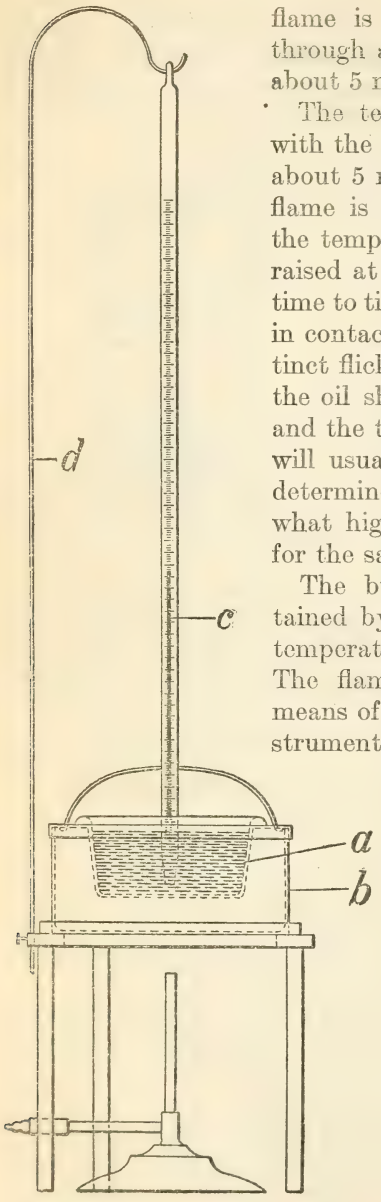
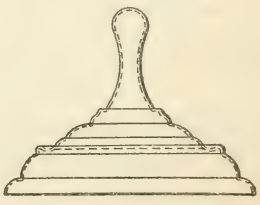


Fig. 10.—Open-cup oil tester.

USE OF FLASH-POINT AND BURNING-POINT DETERMINATIONS.

The flash and burning point determinations should be made on all bituminous road materials which have to be heated before application and upon all fluid and semisolid products



which show a loss by the volatilization test at 163° C. of over 5 per cent. It should also be made upon fluxes which are to be used in cutting hard bitumens.

VOLATILIZATION TEST.

EQUIPMENT.

1 constant-temperature hot-air oven with rubber tubing. (Fig. 11.)
1 thermo-regulator. (Fig. 11–*a*.)
2 chemical thermometers reading from −10° C. to 250° C.
1 tin box, 6 centimeters in diameter by 2 centimeters deep.
1 analytical balance, capacity 100 grams, sensitive to 0.1 milligram.

METHOD.

The object of the volatilization test is to determine the percentage of loss which the material undergoes when 20 grams in a standard-sized container are subjected to a uniform temperature of 163° C. for five hours, and also to ascertain any changes in the character of the material due to such heating.

The oven shown in figure 11, known as the New York Testing Laboratory oven, is used by the Office of Public Roads and Rural Engineering, although any other form may be used that will give a uniform temperature throughout all parts where samples are placed. The bulb of one of the thermometers is immersed in a sample of some fluid, non-volatile bitumen, while the other is kept in air at the same level. The first thermometer serves to show the temperature of the samples during the test, while the latter gives prompt warning of any sudden changes in temperature due to irregularities in the gas pressure, etc.

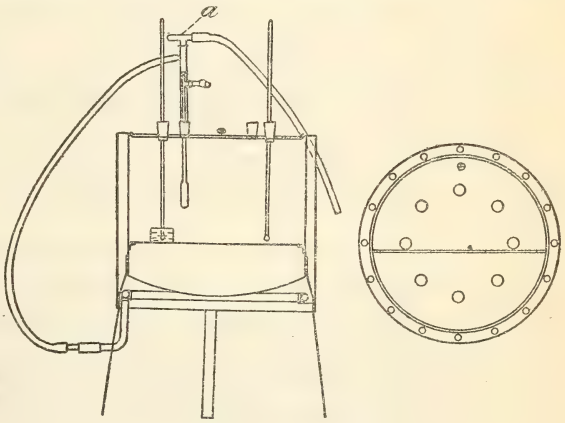


FIG. 11.—New York Testing Laboratory oven.

Before making the test the interior of the oven should show a temperature of 163° C. as registered by the thermometer in air. The tin box is accurately weighed after carefully wiping with a towel to remove any grease or dirt. About 20 grams of the material to be tested is then placed in the box. The material may then be weighed on a rough balance, if one is at hand, after which the accurate weight, which should not vary more than 0.2 gram from the specified amount, is obtained. It may be necessary to warm some of the material in order to handle it conveniently, after which it must be allowed to cool before determining the accurate weight.

The sample should now be placed in the oven, where it is allowed to remain for a period of five hours, during which time the temperature as shown by the thermometer in bitumen should not vary at any time more than 2° C. from 163° C. The sample is then removed from the oven, allowed to cool, and reweighed. From the difference between this weight and the total weight before heating, the percentage of loss on the amount of material taken is calculated.

The general appearance of the residue should be noted, especially with regard to any changes which the material may have undergone. Some relative idea of the amount of hardening which has taken place may be obtained from the results of a float or penetration test made on the residue, as compared with the results of the same test on the original sample. It is also frequently desirable to make the specific gravity and other tests on the residue for the purpose of identifying or ascertaining the character of the base used in the preparation of cut-back products. Before any tests are made on the residue, it should be melted and thoroughly stirred while cooling.

Highly volatile and nonvolatile materials should not be subjected to this test at the same time in the same oven owing to a tendency on the part of the latter to absorb some of the volatile products of the former.

USE OF THE VOLATILIZATION TEST.

The volatilization test, as above described, is made on practically all bitumens with the exception of tars, for which the distillation test answers a similar purpose. The test is also frequently made at 105° C. for five hours, and with products containing small amounts of water it is usually necessary to make a test at the lower temperature before the material can be heated at 163° C. without foaming over. In the case of emulsions it is customary to determine the loss on a 20-gram sample at room temperature for 24 hours, after which the sample is heated at 105° C. for five hours. This additional loss is obtained and all determinations are made on the dried residue and reported accordingly.

The volatilization test is also occasionally made at 205° C. for five hours on a fresh sample in order to show the effect of this higher temperature as compared with the results at 163° C.

Because of the fact that after the volatilization test it frequently happens that a penetration test can not be made upon the residue of a 20-gram sample in the container specified, it has been suggested that the volatilization test be made upon a 50-gram sample in a tin box 5½ centimeters in diameter and 3½ centimeters in depth. In many cases, however, the percentage loss by volatilization and the consequent hardening will be found to vary materially from that obtained with a 20-gram sample owing to differences in the ratio of exposed surface area to total volume of material. This fact should be borne in mind if the test is made with a 50-gram sample.

DISTILLATION TEST.

EQUIPMENT.

1 250 cubic centimeter Engler distillation flask.
1 chemical thermometer reading from 0° C. to 400° C.
1 short condenser, with rubber tubing.
6 25 cubic centimeter glass cylinders, graduated to 0.2 cubic centimeter.
1 iron ring support (ring 7.5 cm. in diameter).
1 iron tripod.
1 burette clamp.
1 tin shield.
1 pinchcock.
2 Bunsen burners, with rubber tubing.
1 quart tin cup, seamless.
1 pint tin cup, seamless.
1 rough balance, capacity 1 kilogram, sensitive to 0.1 gram.
1 analytical balance, capacity 100 grams, sensitive to 0.1 milligrams.

METHOD.

The Engler flasks for this test should meet the following requirements:

Diameter of bulb	8.0 c. m.
Length of neck	15.0 c. m.
Diameter of neck	1.7 c. m.
Length of tubulature	15.0 c. m.
Diameter of tubulature	0.9 c. m.
Angle of tubulature	75°

A 3 per cent variation from the above requirements is allowed.

Thermometers should be thoroughly annealed and filled with carbon dioxide or nitrogen under pressure. The mercury column should rise from 15° to 95° in not more than five seconds, when plunged into boiling water.

The thermometers are calibrated by setting up the entire apparatus (fig. 12) as though a distillation of tar were to be made. One hundred cubic centimeters of material of known boiling point is placed in the flask, which is then heated until the contents distil over at a uniform rate and the thermometer indicates a constant temperature, which is noted. By using three different materials of widely varying boiling point, three calibrations on the thermometer scale are obtained from which other intermediate points may be plotted. The correct fractionating points for the calibrated thermometer are then ascertained.

For calibrating thermometers in the laboratories of the Office of Public Roads and Rural Engineering the fractionating points are obtained from the distillation of distilled water (boiling point 100° C.), chemically pure naphthalene (boiling point 218.2° C.), and chemically pure benzophenone (boiling point 305° C.). The boiling point, of course, varies with the barometric pressure, but if the thermometers are calibrated at a time when the barometer indicates about the

average pressure for a given laboratory, the variations in results due to varying pressures when the thermometer is afterwards used for distillation will be no greater than the possible errors in distillation.

The method as a whole is practically the same as that tentatively recommended in 1911 by the Committee on Standard Tests for Road Materials of the American Society for Testing Materials.[1] Briefly described, it consists in distilling 100 cubic centimeters of the refined or dehydrated tar in an Engler flask at a uniform rate of 1 cubic centimeter per minute and collecting the various fractions in weighed glass graduates. In preparing for the test it will be found convenient to mark permanently on the foot of each graduate its weight to within 0.1 gram.

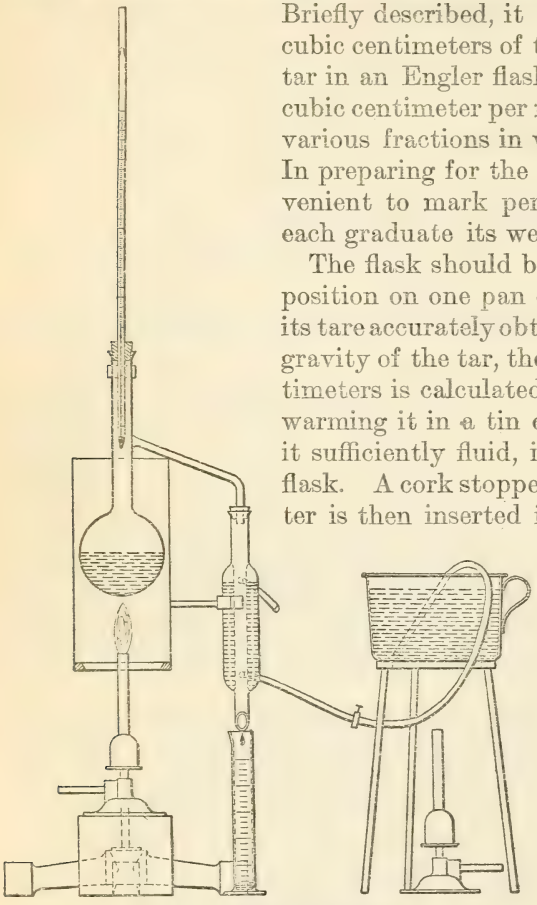


FIG. 12.—Distillation apparatus.

The flask should be supported in a vertical position on one pan of the rough balance and its tare accurately obtained. From the specific gravity of the tar, the weight of 100 cubic centimeters is calculated, and this amount, after warming it in a tin cup, if necessary to make it sufficiently fluid, is poured into the tared flask. A cork stopper carrying the thermometer is then inserted in the neck of the flask, so that the top of the bulb is opposite the middle of the tubulature, and the entire apparatus set up as shown in figure 12. A tin shield with small sight hole surrounds the flask and burner as shown in order to obviate the influence of drafts.

The tar should be heated gradually by means of a Bunsen burner, and the heat should be so regulated as to maintain distillation at the constant rate of 1 cubic centimeter per minute. When the thermometer registers a temperature corresponding to 110° C., the graduated cylinder containing the first fraction is replaced by another. The receiver is changed again at 170° C. and at 270° C., using as many graduated cylinders as may be necessary without allowing any to become filled above the 25-cubic-centimeter mark.

[1] Proc. Am. Soc. for Testing Materials, 1911, Vol. XI, p. 240.

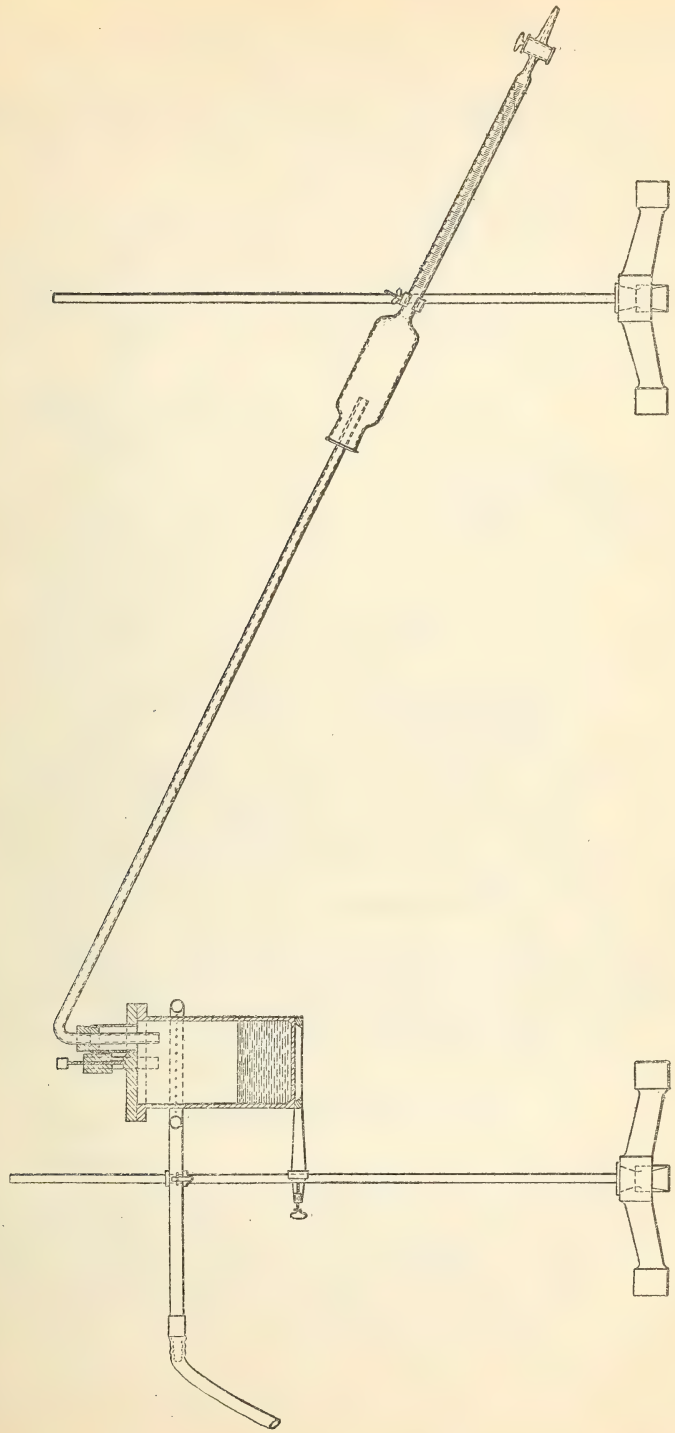


FIG. 13.—Dehydrating apparatus.

When solid matter deposits upon the sides of the condenser, it may be melted by syphoning hot water through the condenser, and collected in the fraction to which it belongs. The last fraction is collected up to 300° C., after which the flask and graduates are cooled to room temperature, and their contents determined by volume and weight. The volume of pitch remaining in the retort is found by deducting the total volume of the distillates from the original 100 cubic centimeters taken. Note should be made of the approximate volume of solids which precipitate from the distillates upon cooling to 25° C.

The results obtained are calculated in percentages by volumes and weights to tenths of 1 per cent and reported as follows:

Distillate.	Per cent, by volume.	Per cent, by weight.
1. Water or ammoniacal liquor		
2. First light oils to 110° C		
3. Second light oils 110° C. to 170° C		
4. Heavy oils 170° C. to 270° C		
5. Heavy oils 270° C. to 300° C		
6. Pitch residue		

The above is applicable only to tars which contain no water. In distilling crude tars or tars which are contaminated with water, it is necessary to dehydrate them before submitting them to the regular distillation. A cylindrical copper still with circular burner, as shown in figure 13, is convenient for this purpose. Two hundred and fifty cubic centimeters of the tar to be dehydrated is weighed in the retort and the apparatus is set up as shown. A low flame is applied to the upper part of the retort and the heating slowly and carefully continued until the volume of water in the separatory funnel shows no further increase. The volume of water collected is noted and calculated. The water is then drained from the separatory funnel and the supernatant layer of oil is run into and thoroughly mixed with the contents of the retort, which should first be cooled below 100° C. A 100 cubic centimeter sample of the dehydrated tar is then submitted to the regular distillation test as above described

USE OF THE DISTILLATION TEST.

The distillation test is made upon tars and tar products but seldom upon other materials unless the presence of tar is suspected, or where a determination of water is required. In making the water determination on viscous or semisolid bituminous materials, it is usually advisable to render the samples fluid by the addition of kerosene or benzol before attempting the distillation.

DIMETHYL SULPHATE TEST.

EQUIPMENT.

Same as specified under distillation test, and in addition: 3 10-cubic centimeter glass cylinders with ground-glass stoppers, graduated to 0.2 cubic centimeter.

METHOD.

The dimethyl sulphate test is employed to detect the presence of petroleum or asphalt products in tar. The pitch (above 300° C.) obtained from the distillation test is used. This pitch, after being cooled and weighed, is again distilled. Fractions are taken at 350° C. and 375° C. These fractions, together with the 270–300° C. fraction previously obtained, are separately stirred and, if necessary, heated to dissolve solids which may be present.

Four cubic centimeters of distillate from each fraction are separately shaken with 6 cubic centimeters of dimethyl sulphate in a 10-cubic centimeter cylinder. After standing 30 minutes the resulting supernatant layer of insoluble oil, from the petroleum or asphalt, is read and calculated to its percentage by volume of the sample of distillate taken. The results are reported as follows:

Fractions.	Per cent of distillate.	Per cent of distillate insoluble in dimethyl sulphate.
270° to 300° C.		
300° to 350° C.		
350° to 375° C.		

USE OF DIMETHYL SULPHATE TEST.

The dimethyl sulphate test is used only in cases where a mixture of petroleum or asphalt products with tar has been specified or is suspected. The test is mainly qualitative, but is valuable when as little as 3 per cent of petroleum or asphalt products are present in the tar.

DETERMINATION OF BITUMEN SOLUBLE IN CARBON DISULPHIDE.

EQUIPMENT.

1 100 cubic centimeter Erlenmeyer flask.
1 500 cubic centimeter flask with side neck for filtering under pressure.
1 rubber stopper with one hole.
1 filter tube, 3.9 centimeters, inside diameter.
1 platinum or porcelain Gooch crucible.
1 piece of seamless rubber tubing, about 3 centimeters in diameter and 3 centimeters long.
50 grams of long-fiber amphibole asbestos.

2 wash bottles; 1 for solvent, 1 for water.
1 Bunsen burner.
1 nichrome triangle.
1 iron tripod.
1 drying oven.
1 desiccator with calcium chloride.
1 thermometer reading from —10° C. to 110° C.
1 vacuum pump and connections.
1 analytical balance, capacity 100 grams, sensitive to 0.1 milligram.

METHOD.

This test consists in dissolving the bitumen in carbon disulphide and recovering any insoluble matter by filtering the solution through an asbestos felt. The form of Gooch crucible best adapted for the determination is 4.4 centimeters wide at the top, tapering to 3.6 centimeters at the bottom, and is 2.5 centimeters deep.

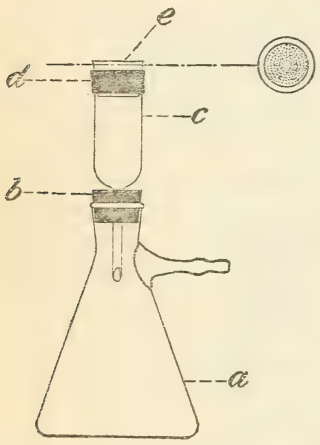


FIG. 14.—Apparatus for determining soluble bitumen.

For preparing the felt the necessary apparatus is arranged as shown in figure 14, in which *a* is the filtering flask, *b* a rubber stopper, *c* the filter tube, and *d* a section of rubber tubing which tightly clasps the Gooch crucible *e*. The asbestos is cut with scissors into pieces not exceeding 1 centimeter in length, after which it is shaken up with just sufficient water to pour easily. The crucible is filled with the suspended asbestos, which is allowed to settle for a few moments. A light suction is then applied to draw off all the water and leave a firm mat of asbestos in the crucible. More of the suspended material is added, and the operation is repeated until the felt is so dense that it scarcely transmits light when held so that the bottom of the crucible is between the eye and the source of light. The felt should then be washed several times with water, and drawn firmly against the bottom of the crucible by an increased suction. The crucible is removed to a drying oven for a few minutes, after which it is ignited at red heat over a Bunsen burner, cooled in a desiccator and weighed.

From 1 to 2 grams of bitumen or about 10 grams of an asphalt topping or rock asphalt is now placed in the Erlenmeyer flask, which has been previously weighed, and the accurate weight of the sample is obtained. One hundred cubic centimeters of chemically pure carbon disulphide is poured into the flask in small portions, with continual agitation, until all lumps disappear and nothing adheres to the bottom. The flask is then corked and set aside for 15 minutes.

After being weighed, the Gooch crucible containing the felt is set up over the dry pressure flask, as shown in figure 14, and the solution of bitumen in carbon disulphide is decanted through the felt without suction by gradually tilting the flask, with care not to stir up any precipitate that may have settled out. At the first sign of any sediment coming out, the decantation is stopped and the filter allowed to drain. A small amount of carbon disulphide is then washed down the sides of the flask, after which the precipitate is brought upon the felt and the flask scrubbed, if necessary, with a feather or "policeman," to remove all adhering material. The contents of the crucible are washed with carbon disulphide, until the washings run colorless. Suction is then applied until there is practically no odor of carbon disulphide in the crucible, after which the outside of the crucible is cleaned with a cloth moistened with a small amount of the solvent. The crucible and contents are dried in the hot-air oven at 100° C. for about 20 minutes, cooled in a desiccator, and weighed. If any appreciable amount of insoluble matter adheres to the flask, it should also be dried and weighed, and any increase over the original weight of the flask should be added to the weight of insoluble matter in the crucible. The total weight of insoluble material may include both organic and mineral matter. The former, if present, is burned off by ignition at a red heat until no incandescent particles remain, thus leaving the mineral matter or ash, which can be weighed on cooling. The difference between the total weight of material insoluble in carbon disulphide and the weight of substance taken equals the total bitumen, and the percentage weights are calculated and reported as total bitumen, and organic and inorganic matter insoluble, on the basis of the weight of material taken for analysis.

This method is quite satisfactory for straight oil and tar products, but where certain natural asphalts are present it will be found practically impossible to retain all of the finely divided mineral matter on an asbestos felt. It is, therefore, generally more accurate to obtain the result for total mineral matter by direct ignition of a 1-gram sample in a platinum crucible or to use the result for ash obtained in the fixed carbon test. The total bitumen is then determined by deducting from 100 per cent the sum of the percentages of total mineral matter and organic matter insoluble. If the presence of a carbonate mineral is suspected, the percentage of mineral matter may be most accurately obtained by treating the ash from the fixed carbon determination with a few drops of ammonium carbonate solution, drying at 100° C., then heating for a few minutes at a dull red heat, cooling, and weighing again.

When difficulty in filtering is experienced—for instance, when Trinidad asphalt is present in any amount—a period of longer subsidence

than 15 minutes is necessary, and the following method proposed by the Committee on Standard Tests for Road Materials of the American Society for Testing Materials is recommended: [1]

From 2 to 15 grams (depending on the richness in bitumen of the substance) is weighed into a 150-cubic centimeter Erlenmeyer flask, the tare of which has been previously ascertained, and treated with 100 cubic centimeters of carbon disulphide. The flask is then loosely corked and shaken from time to time until practically all large particles of the material have been broken up, when it is set aside and not disturbed for 48 hours. The solution is then decanted off into a similar flask that has been previously weighed, as much of the solvent being poured off as possible without disturbing the residue. The first flask is again treated with fresh carbon disulphide and shaken as before, when it is put away with the second flask and not disturbed for 48 hours.

At the end of this time the contents of the two flasks are carefully decanted off upon a weighed Gooch crucible fitted with an asbestos filter, the contents of the second flask being passed through the filter first. The asbestos filter shall be made of ignited long-fiber amphibole, packed in the bottom of a Gooch crucible to the depth of not over one-eighth of an inch. After passing the contents of both flasks through the filter, the two residues are shaken with more fresh carbon disulphide and set aside for 24 hours without disturbing, or until it is seen that a good subsidation has taken place, when the solvent is again decanted off upon the filter. This washing is continued until the filtrate or washings are practically colorless.

The crucible and both flasks are then dried at 125° C. and weighed. The filtrate containing the bitumen is evaporated, the bituminous residue burned, and the weight of the ash thus obtained added to that of the residue in the two flasks and the crucible. The sum of these weights deducted from the weight of substance taken gives the weight of bitumen extracted.

USE OF TOTAL BITUMEN DETERMINATION.

This determination is made on all classes of bituminous products. In the analysis of tars the organic matter insoluble is commonly known and reported as "free carbon."

DETERMINATION OF BITUMEN INSOLUBLE IN PARAFFIN NAPHTHA.

EQUIPMENT.

The apparatus is the same as for bitumen soluble in carbon disulphide.

METHOD.

This determination is made in the same general manner as the total bitumen determination, except that 100 cubic centimeters of 86° to 88° B. paraffin naphtha, at least 85 per cent distilling between 35° C. and 65° C., is employed as a solvent instead of carbon disulphide. Considerable difficulty is sometimes experienced in breaking up some of the heavy semisolid bitumens; the surface of the material is attacked, but it is necessary to remove some of the insoluble matter in order to expose fresh material to the action of the solvent. It is, therefore, advisable to heat the sample after it is weighed, allowing it to cool in a thin layer around the lower part of

[1] Proc. Am. Soc. for Testing Materials, 1909, Vol. IX, p. 221.

the flask. If difficulty is still experienced in dissolving the material, a rounded glass rod will be found convenient for breaking up the undissolved particles. Not more than one-half of the total amount of naphtha required should be used until the sample is entirely broken up. The balance of the 100 cubic centimeters is then added, and the flask is twirled a moment in order to mix the contents thoroughly, after which it is corked and set aside for 30 minutes.

In making the filtration the utmost care should be exercised to avoid stirring up any of the precipitate, in order that the filter may not be clogged and that the first decantation may be as complete as possible. The sides of the flask should then be quickly washed down with naphtha and, when the crucible has drained, the bulk of insoluble matter is brought upon the felt. Suction may be applied when the filtration by gravity almost ceases, but should be used sparingly, as it tends to clog the filter by packing the precipitate too tightly. The material on the felt should never be allowed to run entirely dry until the washing is completed, as shown by the colorless filtrate. When considerable insoluble matter adheres to the flask no attempt should be made to remove it completely. In such cases the adhering material is merely washed until free from soluble matter, and the flask is dried with the crucible at 100° C. for about one hour, after which it is cooled and weighed. The percentage of bitumen insoluble is reported upon the basis of total bitumen taken as 100.

The difference between the material insoluble in carbon disulphide and in the naphtha is the bitumen insoluble in the latter. Thus, if in a certain instance it is found that the material insoluble in carbon disulphide amounts to 1 per cent and that 10.9 per cent is insoluble in naphtha, the percentage of bitumen insoluble would be calculated as follows:

$$\frac{\text{Bitumen insoluble in naphtha}}{\text{Total bitumen}} = \frac{10.9-1}{100-1} = \frac{9.9}{99} = 10 \text{ per cent}$$

USE OF NAPHTHA INSOLUBLE BITUMEN DETERMINATION.

This test is made on all petroleums, malthas, asphalts, and other solid native bitumens and their products.

It should be noted that petroleum naphthas are by no means definite compounds, but are composed of a number of hydrocarbons which vary in character and quantity according to the petroleum from which they have been distilled. Their solvent powers also vary greatly. Thus naphthas produced from asphaltic petroleums, consisting mainly of naphthene and polymethylene hydrocarbons, are much more powerful solvents of the heavier asphaltic hydrocarbons than are the paraffin naphthas. The density of the naphtha also affects its solvent power, for those of high specific gravity dissolve the

heavier hydrocarbons more readily than those of lower specific gravity. As the main object of this test is to separate the heavier hydrocarbons of an asphaltic nature from the paraffin hydrocarbons, a paraffin solvent should be employed, and for ordinary purposes a paraffin naphtha of 86° to 88° B. as described has been found to be readily obtainable and fairly satisfactory.

The determination is also frequently made with heavier naphthas, such as 66° B. and 72° B., for the purpose of grading the character of the bitumen present in the compound. A report should therefore always distinctly state the gravity and character of the solvent used.

DETERMINATION OF BITUMEN INSOLUBLE IN CARBON TETRACHLORIDE.

EQUIPMENT.

The apparatus is the same as for bitumen soluble in carbon disulphide.

METHOD.

This determination is conducted in exactly the same manner as described under "Determination of bitumen soluble in carbon disulphide," using 100 cubic centimeters of chemically pure carbon tetrachloride in place of carbon disulphide.

The percentage of bitumen insoluble is reported upon the basis of total bitumen taken as 100, as described under "Determination of bitumen insoluble in paraffin naphtha."

USE OF DETERMINATION OF BITUMEN INSOLUBLE IN CARBON TETRACHLORIDE.

The bitumen insoluble in carbon tetrachloride, but soluble in carbon disulphide, is commonly known as "carbenes." The test is occasionally made on petroleums, asphalts, and other solid native bitumens and their products, for the purpose of identification, or when there is any reason to suspect that the material under examination has been injured by overheating during the process of manufacture.

DETERMINATION OF FIXED CARBON.

EQUIPMENT.

1 iron ring support (ring 7.5 cm. in diameter).
1 platinum or nichrome triangle.
1 Bunsen burner and rubber tubing.
1 platinum crucible with a tight-fitting cover (weight complete, from 20 to 30 grams).
1 crucible tongs.
1 desiccator with calcium chloride.
1 analytical balance, capacity 100 grams, sensitive to 0.1 milligram.

METHOD.

This determination is made in accordance with the method described for coal in the Journal of the American Chemical Society,

1899, volume 21, page 1116. One gram of the material is placed in a platinum crucible weighing from 20 to 30 grams and having a tightly fitting cover. It is then heated for seven minutes over the full flame of a Bunsen burner, as shown in figure 15. The crucible should be supported on a platinum triangle with the bottom from 6 to 8 centimeters above the top of the burner. The flame should be fully 20 centimeters high when burning freely, and the determination should be made in a place free from drafts. The upper surface of the cover should burn clear, but the under surface should remain covered with carbon, excepting in the case of some of the more fluid bitumens, when the under surface of the cover may be quite clean.

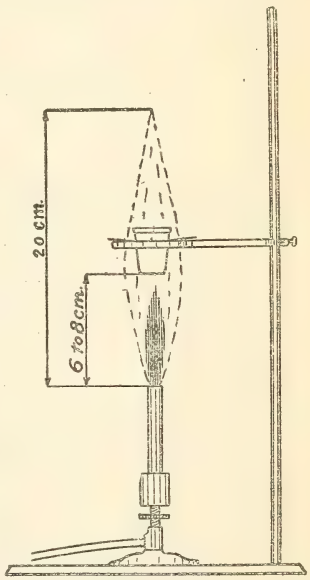


FIG. 15.—Apparatus for determining fixed carbon.

The crucible is removed to a desiccator and when cool is weighed, after which the cover is removed, and the crucible is placed in an inclined position over the Bunsen burner and ignited until nothing but ash remains. Any carbon deposited on the cover is also burned off. The weight of ash remaining is deducted from the weight of the residue after the first ignition of the sample. This gives the weight of the so-called fixed or residual carbon, which is calculated on a basis of the total weight of the sample, exclusive of mineral matter. If the presence of a carbonate mineral is suspected, the percentage of mineral matter may be most accurately obtained by treating the ash with a few drops of ammonium carbonate solution, drying at 100° C., then heating for a few minutes at a dull red heat, cooling and weighing.

An excellent form of crucible for this test is shown in figure 15. It has a cover with a flange 4 millimeters wide, fitting tightly over the outside of the crucible, and weighs complete about 25 grams. Owing to sudden expansion in burning some of the more fluid bitumens, it is well to hold the cover down with the end of the tongs until the most volatile products have burned off.

Some products, particularly those derived from Mexican petroleum, show a tendency to suddenly expand and foam over the sides of the crucible in making this determination, and no method of obviating this trouble without vitiating the result has thus far been forthcoming. Recent experiments in the laboratory of the Office of

Public Roads and Rural Engineering indicate that the difficulty may be overcome by placing a small piece of platinum gauze over the sample and about midway of the crucible. The gauze should be so cut or bent as to touch the sides of the crucible at all points, and is of course weighed in place in the crucible before and after ignition.

USE OF DETERMINATION FOR FIXED CARBON.

This determination is made on all bituminous products with the exception of tars, upon which reliable results can not be obtained, owing to the error introduced by the presence of considerable quantities of free carbon.

DETERMINATION OF PARAFFIN SCALE.

EQUIPMENT.

1 one-half pint iron retort. (Fig. 16.)
1 piece iron tubing, 30 inches long.
2 100 cubic centimeter Erlenmeyer flasks.
1 500 cubic centimeter (16 oz.) flask, with side neck for filtering under pressure.
1 freezing apparatus. (Fig. 17.)
1 6-inch test tube, ¾-inch diameter.
1 analytical balance, capacity 100 grams, sensitive to 0.1 milligram.
1 rough balance, capacity 1 kilogram, sensitive to 0.1 gram.
1 wash bottle.
1 pint tin cup, seamless.
1 vacuum pump and connections.
1 glass crystallizing dish, 50 millimeters in diameter.
1 steam bath.
1 desiccator with calcium chloride.
1 4-inch steel spatula.
1 Bunsen burner with rubber tubing.
2 iron stands with retort clamp, and 1 ring.

METHOD.

Fifty grams of the material under examination should be weighed into the tared iron retort and distilled as rapidly as possible to dry coke. The distillation should be complete in not over 25 minutes. The distillate is caught in a 100-cubic centimeter Erlenmeyer flask, the weight of which has been previously ascertained. During the early stages of distillation a cold, damp towel wrapped around the stem of the retort will serve to condense the distillate. After high temperatures have been reached, this towel may be removed. When the distillation is completed the distillate is allowed to cool to room temperature and is then weighed in the flask. This weight minus that of the flask gives the weight of the total distillate.

The apparatus for freezing out and separating the paraffin scale is shown in figure 17. It consists of a bell jar *a* about 16 centimeters high and 14 centimeters in diameter, surrounded by a felt or cotton

cover *b*. A copper jacket *c*, 4½ centimeters in diameter at the top and 21 centimeters long, is held in the neck of the bell jar by means of a rubber stopper *d*, and fits into the upper portion of the rubber stopper *j*. A glass filter tube *e* fits inside the copper jacket, and to prevent circulation of air and condensation of water between it and the jacket, a strip of heavy blotting paper *f* is wrapped around the top of the filter tube. Just below the constriction in the filter tube a wad of absorbent cotton *g* is placed, tightly compressed to a length of 2 centimeters by means of a glass rod. Above this is a wad of tightly packed asbestos wool *h*, about 5 millimeters in length, upon which an asbestos filtering mat *i* is prepared. The filter tube passes through a rubber stopper *j* into a vacuum filtering flask *k* of 500 cubic centimeters capacity. The rubber stopper *j* is placed as tightly as possible against the neck of the bell jar, but to insure that there is no circulation of air, a disk of blotting paper *l* is compressed between them. The thermometer *m* is capable of recording temperatures from −25° C. to 0° C. and has the −20° C. graduation at least 14 centimeters from the bulb. It is supported by means of a guiding cork *n* and cork disk *o*, which is held tightly against the top of the filter tube by the clamp *p*.

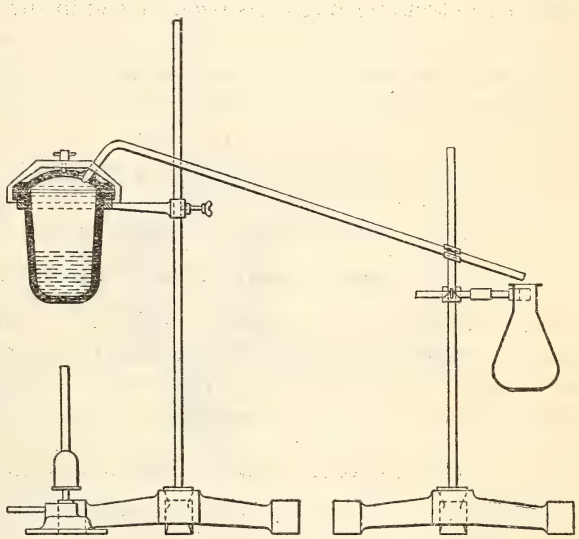


FIG. 16.—Apparatus for distillation in determination of paraffin scale.

The bell jar is filled with a freezing mixture of ice and salt in the proportion of three to one, which as it melts is drawn off through the bent glass tube *q*, which is fitted with a rubber connection *r* and pinchcock *s* and collected in the 500 cubic centimeter filtering flask *t*. The apparatus is supported on the ring *u* and condenser clamp *v*, attached to the stand *w*.

In separating the paraffin scale the following procedure is carried out. The filtering flask *k* is removed and a small cork stopper inserted in the lower end of the filter tube to assist in retaining the solution to be chilled in the upper part of the tube. Ten cubic centimeters of a mixture of equal parts Squibbs' ether and absolute alcohol

is poured into the filter tube, the temperature of which has been reduced to −20° C. From one to two grams of the well-mixed distillate obtained in the manner previously described is then accurately weighed in a 100 cubic centimeter Erlenmeyer flask, mixed with 10 cubic centimeters of Squibbs' ether, and poured into the filter tube. Ten cubic centimeters of absolute alcohol is next placed in the flask to wash out the ether solution, poured into the filter tube, and the cover carrying the thermometer placed on the tube. The mixture is maintained at a temperature of −20° C. for 15 minutes, then the cork stopper is removed from the outlet of the filter tube and the filtering flask is replaced. The corks supporting the thermometer are now loosened and a strong suction is applied to the filter flask until all of the solvent is drawn off. The contents of the filter tube are next washed with 10 cubic centimeters of a 1 to 1 mixture of Squibbs' ether and absolute alcohol, which is chilled to −20° C. in the filter tube before suction is applied. When the washings have been removed the vacuum is turned off and the filter tube removed from the apparatus. The filter tube is then placed in a clean filter flask which also contains a 6-inch test tube in which the dissolved paraffin scale is later collected. About 10 cubic centimeters of warm petroleum ether is poured into the filter tube and allowed to remain until the paraffin scale has been dissolved. Vacuum is then applied and the dissolved scale drawn into the test tube. This treatment is followed by two washings, one of 10 cubic centimeters and the other of 5 cubic centimeters of warm petroleum ether, which removes the last traces of paraffin scale. The entire contents of the test tube are

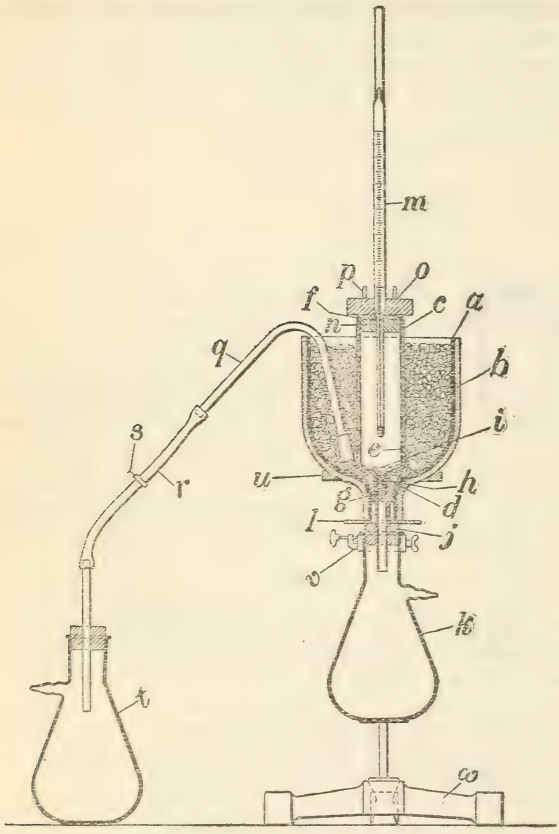


FIG. 17.—Freezing apparatus for determining paraffin scale.

then poured into a weighed platinum or glass crystallizing dish and the petroleum ether evaporated off over a steam bath. The dish is then placed in a drying oven maintained at 105° C. until the last traces of petroleum ether have been removed and the paraffin scale has attained a constant weight, after cooling in a desiccator.

The weight of the paraffin scale so obtained, divided by the weight of the distillate taken and multiplied by the percentage of the total distillate obtained from the original sample, equals the percentage of the paraffin scale.

USE OF PARAFFIN SCALE DETERMINATION.

The paraffin scale determination may be made on all native bitumens and their products which are suspected of being of a paraffin nature. It is not an extremely accurate determination, however, and is seldom employed by the Office of Public Roads and Rural Engineering.

THE EXTRACTION OF BITUMINOUS AGGREGATES.

EQUIPMENT FOR RECOVERING AGGREGATE ONLY.

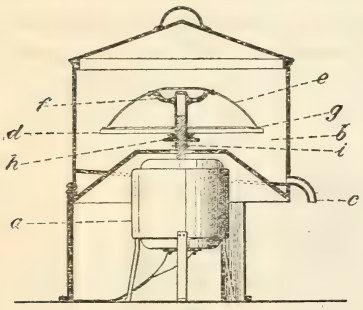
1 centrifuge extractor, complete with motor, speed regulator, and electrical connections. (Fig. 18.)
1 hot plate.
1 enamel-ware dish approximately 2 inches deep and 9 inches in diameter.
1 hammer.
1 three-fourths-inch cold chisel.
1 large metal kitchen spoon.
1 square foot of one-sixteenth-inch deadening felt paper.
1 1½-inch stiff flat brush.
1 500-cubic-centimeter bottle or flask.
1 balance, capacity 1 kilogram, sensitive to 0.1 gram.
1 sheet of heavy manila paper.

ADDITIONAL EQUIPMENT FOR RECOVERING BITUMEN.

1 iron ring support (ring 10 centimeters in diameter).
1 iron ring support with condenser clamp.
1 round tin can, 10 by 12 centimeters, covered with asbestos paper.
1 100-watt incandescent carbon-filament lamp, with socket and connections.
1 asbestos hood. (Fig. 19–c.)
1 1,000-cubic-centimeter round-bottom flask, with cork.
1 spiral condenser, length of body 25 centimeters, with cork to fit, and rubber-tubing connections.
50 centimeters of glass tubing, 8 millimeters bore.
1 1,000-cubic-centimeter flat-bottom flask.
1 porcelain evaporating dish, 11 centimeters in diameter.
1 watch glass, 20 centimeters in diameter.
1 steam bath.

METHOD.

The extractor shown in figure 18 was designed upon lines suggested by an examination of machines in use by A. E. Schutte and C. N. Forrest. It consists of a one-fifth-horsepower 1,100 revolutions per minute vertical-shaft electric motor *a*, with the shaft projecting into the cylindrical copper box *b*, the bottom of which is so inclined as to drain to the spout *c*. A three-sixteenths-inch circular brass plate $9\frac{1}{2}$ inches in diameter is shown in *d*, and upon this rests the sheet-iron bowl *e*, which is $8\frac{1}{2}$ inches in diameter by $2\frac{5}{16}$ inches high, and has a 2-inch circular hole in the top. Fastened to the inner side of the bowl is the brass cup *f*, having a circle of one-eighth-inch holes for the admission of the solvent, and terminating in the hollow axle, which fits snugly through a hole at the center of the brass plate. The bowl may be drawn firmly against a felt-paper ring *g*, three-fourths inch wide, by means of the $2\frac{1}{2}$-inch milled nut *h*, for which the hollow axle is threaded for a distance of three-fourths inch directly below the upper surface of the plate. The axle fits snugly over the shaft of the motor, to which it is locked by a slot and cross pin *i*.



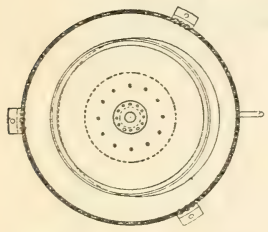


FIG. 18.—Centrifuge extractor (Reeve type).

The aggregate is prepared for analysis by heating it in an enamel-ware pan on the hot plate until it is sufficiently soft to be thoroughly disintegrated by means of a large spoon. Care must be taken, however, that the individual particles are not crushed. If a section of pavement is under examination, a piece weighing somewhat over 1 kilogram may be cut off with hammer and chisel. The disintegrated aggregate is then allowed to cool, after which a sufficient amount is taken to yield on extraction from 50 to 60 grams of bitumen. It is placed in the iron bowl and a ring three-fourths of an inch wide, cut from the felt paper, is fitted on the rim, after which the brass plate is placed in position and drawn down tightly by means of the milled nut. If the bitumen is to be recovered and examined, the felt ring should be previously treated in the empty extractor with a couple of charges of carbon disulphide in order to remove any small amount of grease or resin that may be present, although a proper grade of felt should be practically free from such products. The bowl is now placed on the motor shaft and the slot and pin are carefully locked. An empty bottle is placed under the spout and 150 cubic centimeters of carbon

disulphide is poured into the bowl through the small holes. The cover is put on the copper box and, after allowing the material to digest for a few minutes, the motor is started, slowly at first in order to permit the aggregate to distribute uniformly. The speed should then be increased sufficiently by means of the regulator to cause the dissolved bitumen to flow from the spout in a thin stream. When the first charge has drained, the motor is stopped and a fresh portion of disulphide is added. This operation is repeated from four to six times with 150 cubic centimeters of disulphide. With a little experience the operator can soon gauge exactly what treatment is necessary for any given material. When the last addition of solvent has drained off, the bowl is removed and placed with the brass plate uppermost on a sheet of manila paper. The brass plate and felt ring are carefully laid aside on the paper and, when the aggregate is thoroughly dry, it can be brushed on a pan of the rough balance and weighed. The difference between this weight and the original weight taken shows the amount of bitumen extracted. The aggregate may then be tested as occasion requires.

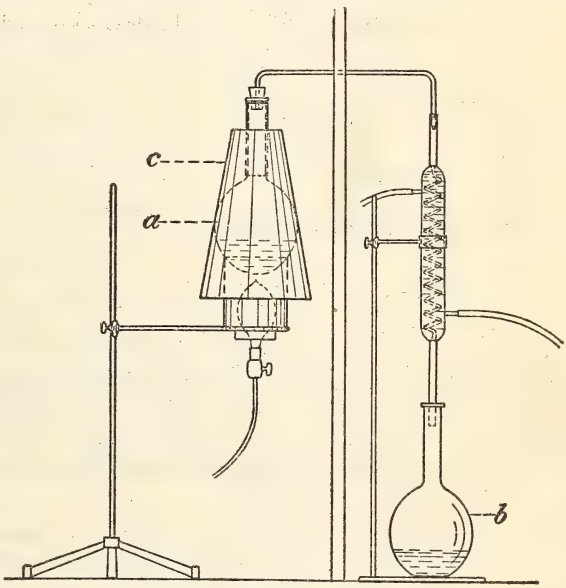


FIG. 19.—Recovery apparatus.

When it is desired to recover and examine the bitumen, the apparatus shown in figure 19 will be found convenient and fairly safe for the distillation and recovery of such inflammable solvents as carbon disulphide. In the laboratory of the Office of Public Roads and Rural Engineering this apparatus is arranged so that the glass tubing passes through a stone partition between two sections of a small hood, thus keeping the distilling and receiving apparatus entirely separated.

The solution of bitumen should be allowed to stand overnight in order to permit the settling of any fine mineral matter that is sometimes carried through the felt ring in the extractor. The solution is then decanted into the flask *a*, and the solvent is driven off by means of heat from an incandescent lamp until the residue is of a thick sirupy consistency. Meanwhile the solvent is condensed and recovered in the flask *b*. The residue is poured into an 11-centimeter

porcelain evaporating dish and evaporated on a steam bath. The most scrupulous care must be taken at all times that no flames are in its immediate vicinity. Evaporation is carried on at a gentle heat, with continual stirring, until foaming practically ceases. It is advisable to have a large watch glass at hand to smother the flames quickly should the material ignite. As the foaming subsides, the heat of the steam bath may be gradually raised, and evaporation is continued until the bubbles beaten or stirred to the surface of the bitumen fail to give a blue flame or odor of sulphur dioxide when ignited by a small gas jet. The dish of bitumen should then be set in a hot-air oven maintained at 105° C. for about an hour, after which it is allowed to cool. Its general character is noted and any tests for bitumens that are necessary are then made upon it.

GRADING THE MINERAL AGGREGATE.

EQUIPMENT.

1 set of 8-inch stone sieves with circular openings of 1½, 1¼, 1, ¾, ½, and ¼ inches, respectively.
1 set of 8-inch brass sand sieves of 10, 20, 30, 40, 50, 80, 100, and 200 mesh, respectively, with pan and cover.
1 rough balance, capacity 1 kilogram, sensitive to 0.1 gram.
1 1½-inch stiff flat brush.
Several sheets of manila paper.

METHOD.

While a mechanical sifter is employed in the Office of Public Roads and Rural Engineering, the following hand method is given for the benefit of those who have not a machine of this character available. When a machine is used its method of operation should be checked against the hand method described below to obtain practically equivalent results.

For aggregates containing particles too large to pass a 10-mesh screen, the stone sieves are used, and are stacked in their regular order over a sheet of heavy paper, with the largest size required on top. The weighed amount of stone is placed on the largest sieve and is carefully protected from drafts which might carry away any of the fine material. The upper sieve is then removed from the stack and shaken over a large sheet of paper until no more particles come through. The material thus retained, including any fragments caught in the meshes of the sieve, is weighed and that which passes is added to the contents of the succeeding sieve. This operation is repeated with each succeeding sieve.

When grading sands or fine aggregates, it is customary to take a 100-gram sample in order that the weights may give direct percentages to tenths of 1 per cent. The sieves are stacked in regular order with the 200-mesh sieve resting on the pan. The sample is brushed on the

top sieve, after which the cover is put on and the stack agitated for about five minutes with both rocking and circular shaking. Each sieve is removed in order, and shaken and tapped on a clean piece of paper until no appreciable amount of material comes through. All lumps are broken up by crushing them against the side of the sieve with the finger or a small spatula. The contents of the sieve are emptied into the pan of the balance. All particles caught in the mesh are removed by brushing across the underside of the sieve and are added to the contents of the pan. As great opportunity exists for wide variations in the results of sand gradings made by different persons, owing to the possibility of always getting a little more material to pass by continued shaking, it is well for the novice to repeat his sifting on any given mesh, after having weighed it, in order to see what further loss he can produce. If his judgment has not erred, several minutes' further sifting should not produce a loss of over 0.5 gram.

Where coarse aggregates have considerable material passing a 10-mesh sieve and it is desired to grade this material further, it should be weighed and well mixed, quartered, if necessary, and a 100-gram sample should be passed through the sand sieves. From the percentages so obtained and the weight of material passing the 10-mesh sieve, the percentages of the total aggregate which these finer materials represent may be calculated.

The Office of Public Roads and Rural Engineering has adopted the following recommendations of the Committee on Standard Tests for Road Materials of the American Society for Testing Materials as to the size of wire for standard sand sieves:

Meshes per linear inch:	Diameter of wire, inches.	Meshes per linear inch:	Diameter of wire, inches.
10	0.027	50	0.009
20	.0165	80	.00575
30	.01375	100	.0045
40	.01025	200	.00235

DETERMINATION OF VOIDS IN THE MINERAL AGGREGATE.

EQUIPMENT.

1 1,000-cubic-centimeter graduated glass cylinder.
1 500-cubic-centimeter graduated glass cylinder.
2 pieces brass tubing (¼-inch bore and 2 inches long).
1 wooden block mounted with soft rubber pad.
1 iron support with condenser clamp.
3 feet rubber tubing.
1 pinchcock.
1 1½-inch stiff flat brush.
1 small tin scoop.
2 sheets of manila paper.

METHOD.

The cylinders are constructed for this purpose by drilling a hole in the center of the bottom of each. This hole should be slightly larger than the outside diameter of the brass tubing, one end of which is cemented into it by means of a litharge and glycerin mixture. The upper end of the tube should be flush with the inside of the bottom of the graduated cylinder and in the large cylinder a piece of 200-mesh wire gauze should be soldered over the end of the tube to prevent fine material passing through.

The apparatus is set up as shown in figure 20, with the pinchcock on the rubber tubing closed. The small cylinder is filled with kerosene, after which the pinchcock is slowly opened in order to permit the kerosene to force any air from the tubing and to come flush with the bottom of the large cylinder. The pinchcock is then closed.

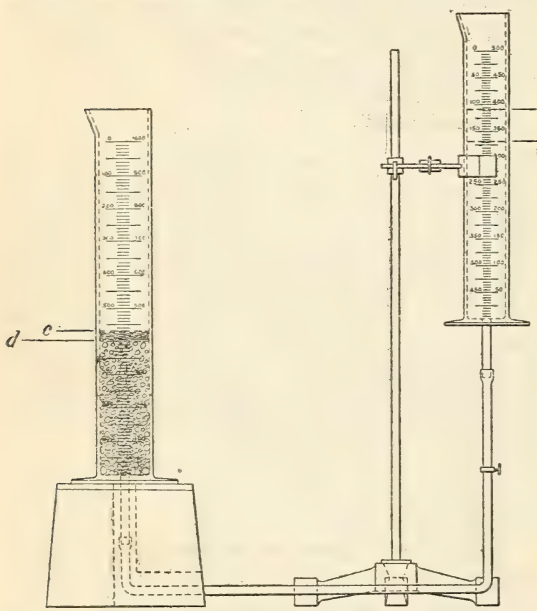


FIG. 20.—Apparatus for determining voids in the mineral aggregate.

The aggregate is thoroughly mixed and quartered, if necessary, until a representative sample of material of at least 300 cubic centimeters volume is obtained. This sample of aggregate is poured into the large cylinder, a scoopful at a time, with a light tamping of the cylinder against the rubber pad in order to compact the material. Best results are usually obtained by making a cylinder of manila paper inside the glass cylinder, and introducing the aggregate inside the paper. The paper can then be slowly withdrawn while the glass cylinder is being lightly tamped. Segregation of the several sizes of material must be avoided, and any fine material remaining on the paper should be brushed off and added to the aggregate.

When the aggregate in the large cylinder has reached its maximum state of compaction, the volume of kerosene in the small cylinder is read, the pinchcock is opened and the elevation of the small cylinder so regulated as to permit the kerosene to slowly percolate upward through the aggregate, until it has reached a point 20 or 30 cubic centi-

meters above the top of the aggregate. The meniscus of the kerosene in each cylinder and the volume of aggregate is then noted. The percentage of voids is calculated as follows:

a equals initial volume of kerosene in small cylinder.
b equals final volume of kerosene in small cylinder.
c equals meniscus of kerosene in large cylinder.
d equals apparent volume of aggregate.

Percentage of voids equals $\frac{(a-b)-(c-d)}{d}$

In some cases the kerosene fails to expel all the air from the aggregate, and this fact will be evidenced by bubbles coming to the surface if the aggregate is stirred with a long thin metal rod after the final readings are taken. Accurate results under such conditions are obtained by stirring the aggregate until bubbles cease to appear. This will, of course, yield a lower reading on the meniscus of the kerosene in the large cylinder, but the original reading on the volume of rock should be taken.

METHODS OF EXAMINING BITUMINOUS EMULSIONS.

The exact determination of the constituents of a bituminous emulsion is usually attended with considerable difficulty and no predetermined scheme can be made applicable to all materials of this character. In a number of cases, however, the following method has yielded satisfactory and fairly accurate results.

In order to break up the emulsion, a 20-gram sample is digested on a steam bath with 100 cubic centimeters of $\frac{N}{2}$ alcoholic potash. The digestion is carried out in a flask with a reflux condenser for about 45 minutes. The solution is filtered and the precipitate washed with 95 per cent alcohol. The filtrate is evaporated to dryness, after which the residue is taken up with hot water and any insoluble matter is filtered off. The aqueous solution, which contains the potassium soaps of the fatty acids, is acidified with dilute sulphuric acid and then shaken in a separatory funnel with petroleum ether. The aqueous portion is drawn off and the ethereal layer shaken up with cold water and washed twice, after which it is evaporated in a weighed platinum or porcelain dish to constant weight, first over a steam bath and then in a drying oven at 105° C. The residue consists of the fatty and resin acids present in the emulsion.

The percentage of water in the emulsion is determined by distilling a 100-gram sample in the retort used for the dehydration of tars. The distillation is carried out in exactly the same manner as described for crude tars until the volume of water in the receiver shows no further increase. Any oils that come over are thoroughly mixed with the material remaining in the retort.

A 2-gram sample of this dehydrated material is extracted with carbon disulphide as described in the method for the determination of bitumen soluble in carbon disulphide, and in this manner the organic matter insoluble in carbon disulphide can be determined. A 1-gram sample of the dehydrated material is ignited. The ash will contain any inorganic matter from the bitumen as well as the fixed alkali present in the soap. The results are, of course, all calculated on a basis of the original material.

Many emulsions contain ammonia, and when this is present a second distillation of the material is necessary. This is carried out on a 100-gram sample in exactly the same manner as described for the determinations of water, excepting for the fact that 40 cubic centimeters of a 10 per cent solution of caustic potash is added to the contents of the retort before beginning the distillation. The distillate is collected in a measured volume of $\frac{N}{2}$ sulphuric acid. When the distillation is completed the excess of acid is titrated with $\frac{N}{2}$ caustic potash, and the ammonia thus determined.

Having determined all constituents as above noted, it is assumed that the difference between their sum and 100 per cent is bitumen, which amount is reported accordingly.

APPENDIX.

LABORATORY EQUIPMENT.

The necessary equipment for a small laboratory about to engage in the ordinary routine testing and inspection of bituminous materials is given in the following list. The maximum cost, exclusive of platinum ware, solvents, and chemicals, should not exceed $300, and the material could no doubt be purchased at a lower figure by securing bids on the entire equipment from several of the chemical supply houses.

For the extraction of bituminous aggregates, the recovery of the bitumen, and examination of the aggregates, the additional equipment described for that work will be required at an additional cost of approximately $150.

APPARATUS.

1 analytical balance, capacity 100 grams, sensitive to 0.1 milligram.
1 set of weights, 50 grams to 5 milligrams, with rider.
1 rough balance, capacity 1 kilogram, sensitive to 0.1 gram.
1 stop watch.
1 Engler viscosimeter.
1 penetrometer, with seconds pendulum or metronome.
1 New York State Board of Health oil tester.
1 open cup oil tester with Bunsen burner.
1 aluminum float with 3 brass collars.
1 cubical brass mold.
1 brass plate 5 by 8 centimeters.
1 metal cover for melting point apparatus.
1 constant temperature oven.
1 thermo-regulator.
1 hot plate 14 by 18 inches.
1 steam bath.
1 small drying oven.
6 Bunsen burners.
1 pinchcock.
4 iron tripods.
4 iron ring supports.
3 rings for ring support, 7.5 centimeters in diameter.
3 condenser clamps.
3 burette clamps.
6 pieces of wire gauze, 10 by 10 centimeters.
2 enamel-ware dishes, 3 inches deep by 9 inches in diameter.
1 tin shield.
6 1-pint tin cups, seamless.
6 1-quart tin cups, seamless.
12 tin boxes, 6 centimeters in diameter by 2 centimeters deep.

6 tin boxes, 5 centimeters in diameter by 3.5 centimeters deep.
1 metal kitchen spoon.
1 ½-pint iron retort.
1 piece iron tubing, 30 inches long.
1 copper still, with steel clamps, inside dimensions 6 by 3½ inches.
1 ring burner to fit copper still.
1 10-centimeter steel spatula.
1 crucible tongs.
1 hammer.
1 ¾-inch cold chisel.
1 1½-inch stiff flat brush.
1 triangular file.
1 small round file.
1 set of cork borers, Nos. 1 to 12.
1 brass filter (vacuum) pump.
1 piece of wire, 20 centimeters long (No. 12 Brown & Sharpe gauge).
3 chemical thermometers reading from −10° C. to 110° C.
3 chemical thermometers reading from 0° C. to 250° C.
2 chemical thermometers reading from 0° C. to 400° C.
4 hydrometers, 0.800 to 0.900; 0.900 to 1.000; 1.000 to 1.200; 1.200 to 1.400.
1 hydrometer jar, 350 by 50 millimeters.
1 special pycnometer.
6 250 cubic centimeter beakers, low form.
6 150 cubic centimeter beakers, low form.
2 800 cubic centimeter beakers, low form.
2 400 cubic centimeter beakers, tall form, without lip.
1 10-centimeter crystallizing dish, deep, straight sides.
1 5-centimeter crystallizing dish.
12 100 cubic centimeter Erlenmeyer flasks.
2 150 cubic centimeter Erlenmeyer flasks.
2 500 cubic centimeter flasks with side neck for filtering under pressure.
6 250 cubic centimeter Engler distillation flasks of special dimensions.
1 short condenser.
1 separatory funnel with stopcock.
4 500 cubic centimeter wash bottles, with tubulated glass stopper ground into neck for solvents.
1 1,000 cubic centimeter wash bottle for water.
6 100 cubic centimeter glass cylinders, graduated to 1 cubic centimeter.
6 25 cubic centimeter glass cylinders, graduated to 0.2 cubic centimeter.
3 10 cubic centimeter glass cylinders with ground-glass stoppers graduated to 0.2 cubic centimeter.
1 150-millimeter desiccator with porcelain plate.
1 filter tube for Gooch crucible 3.9 centimeters in diameter.
1 object glass.
1 freezing apparatus.
1 glass connecting tube.
1 pound of light glass tubing, assorted.
1 pound of glass rods, assorted.
2 rubber stoppers, No. 8, with one hole.
2 rubber stoppers, No. 1, with one hole.
5 pounds of rubber gas tubing, ¼ inch internal diameter.
1 foot of rubber tubing, 1 inch diameter, for Gooch crucibles.
5 pounds of heavy asbestos paper.
Corks, assorted.

PLATINUM.

1 triangle, 5 centimeter sides (10 to 12 grams), or 1 nichrome triangle.
1 Gooch crucible, as specified (20 to 25 grams) for solubility determinations (may be porcelain).
1 crucible with tight-fitting, flanged lid (about 25 grams) for fixed carbon determinations.

SOLVENTS AND CHEMICALS

Carbon disulphide, chemically pure.
86° Baumé paraffin naphtha, distilling between 40° C. and 65° C.
Carbon tetrachloride, chemically pure.
Ether (Squibb's).
Alcohol, absolute (Squibb's).
Dimethyl sulphate.
Naphthalene (C. P.).
Benzophenone (C. P.).
Cottonseed oil.
Mercuric chloride or nitrate.
Mercury.
Ammonium carbonate (C. P.).
Calcium chloride, granular for desiccator.
Asbestos, pure long-fiber amphibole.

Metric conversion tables.

Length.			Capacity.		Mass.	
Inches.	Millimeters.		United States liquid ounces.	Cubic centimeters.	Avoirdupois ounces.	Grams.
$\frac{1}{32}$=0.0312	0.7938		1.....	29.574	1.....	28.3495
$\frac{1}{16}$= .0625	1.5875		2.....	59.147	2.....	56.6991
$\frac{1}{8}$= .1250	3.1750		3.....	88.721	3.....	85.0486
$\frac{1}{4}$= .2500	6.3500		4.....	118.295	4.....	113.3981
$\frac{1}{2}$= .5000	12.7000		5.....	147.869	5.....	141.7476
1.....	25.4001		6.....	177.442	6.....	170.0972
2.....	50.8001		7.....	207.016	7.....	198.4467
3.....	76.2002		8.....	236.590	8.....	226.7962
4.....	101.6002		9.....	266.163	9.....	255.1457
5.....	127.0003		16=1 pt...	473.18	16=1 lb....	453.59
6.....	152.4003		32=1 qt...	946.36	0.3527.....	10
7.....	177.8004		128=1 gal...	3,785.43	.7055	20
8.....	203.2004		0.3381.....	10	1.0582.....	30
9.....	228.6005		.6763.....	20	1.4110.....	40
		Centimeters.	1.0144.....	30	1.7637.....	50
			1.3526.....	40	2.1164.....	60
			1.6907.....	50	2.4692.....	70
			2.0288.....	60	2.8219.....	80
			2.3670.....	70	3.1747.....	90
			2.7051.....	80	3.5270.....	100
0.3937	1=10 mm.		3.0432.....	90	35.27.....	1,000=1 kilogram.
.7874	2		3.3810.....	100		
1.1811	3		33.81.....	1,000=1 liter.		
1.5748	4					
1.9685	5					
2.3622	6					
2.7559	7					
3.1496	8					
3.5433	9					

Comparison of degrees Baumé and specific gravity.

(Liquids lighter than water.)

(1) Sp. gr.$=\frac{140}{130+°B.}$ at 15.5° C. (2) °B.$=\frac{140}{\text{Sp. gr.}}-130$ at 15.5° C.

°B.	Sp. gr.	°B.	Sp. gr.	°B.	Sp. gr.	°B.	Sp. gr.
10	1.0000	31	0.8695	52	0.7692	73	0.6896
11	.9929	32	.8641	53	.7650	74	.6863
12	.9859	33	.8588	54	.7609	75	.6829
13	.9790	34	.8536	55	.7567	76	.6796
14	.9722	35	.8484	56	.7526	77	.6763
15	.9655	36	.8433	57	.7486	78	.6731
16	.9589	37	.8383	58	.7446	79	.6698
17	.9523	38	.8333	59	.7407	80	.6666
18	.9459	39	.8284	60	.7368	81	.6635
19	.9395	40	.8235	61	.7330	82	.6604
20	.9333	41	.8187	62	.7292	83	.6573
21	.9271	42	.8139	63	.7254	84	.6542
22	.9210	43	.8092	64	.7216	85	.6511
23	.9150	44	.8045	65	.7179	86	.6482
24	.9090	45	.8000	66	.7143	87	.6452
25	.9032	46	.7954	67	.7107	88	.6422
26	.8974	47	.7909	68	.7071	89	.6393
27	.8917	48	.7865	69	.7035	90	.6363
28	.8860	49	.7821	70	.7000		
29	.8805	50	.7777	71	.6965		
30	.8750	51	.7734	72	.6931		

Comparison of Centigrade and Fahrenheit degrees.

(1) °F.$=\frac{9}{5}$°C.$+32$. (2) °C.$=\frac{5(°F.-32)}{9}$.

C.	F.	C.	F.	C.	F.	C.	F.	C.	F.	C.	F.
0	32.0	38	100.4	76	168.8	114	237.2	152	305.6	190	374.0
1	33.8	39	102.2	77	170.6	115	239.0	153	307.4	191	375.8
2	35.6	40	104.0	78	172.4	116	240.8	154	309.2	192	377.6
3	37.4	41	105.8	79	174.2	117	242.6	155	311.0	193	379.4
4	39.2	42	107.6	80	176.0	118	244.4	156	312.8	194	381.2
5	41.0	43	109.4	81	177.8	119	246.2	157	314.6	195	383.0
6	42.8	44	111.2	82	179.6	120	248.0	158	316.4	196	384.8
7	44.6	45	113.0	83	181.4	121	249.8	159	318.2	197	386.6
8	46.4	46	114.8	84	183.2	122	251.6	160	320.0	198	388.4
9	48.2	47	116.6	85	185.0	123	253.4	161	321.8	199	390.2
10	50.0	48	118.4	86	186.8	124	255.2	162	323.6	200	392.0
11	51.8	49	120.2	87	188.6	125	257.0	163	325.4	201	393.8
12	53.6	50	122.0	88	190.4	126	258.8	164	327.2	202	395.6
13	55.4	51	123.8	89	192.2	127	260.6	165	329.0	203	397.4
14	57.2	52	125.6	90	194.0	128	262.4	166	330.8	204	399.2
15	59.0	53	127.5	91	195.8	129	264.2	167	332.6	205	401.0
16	60.8	54	129.2	92	197.6	130	266.0	168	334.4	206	402.8
17	62.6	55	131.0	93	199.4	131	267.8	169	336.2	207	404.6
18	64.4	56	132.8	94	201.2	132	269.6	170	338.0	208	406.4
19	66.2	57	134.6	95	203.0	133	271.4	171	339.8	209	408.2
20	68.0	58	136.4	96	204.8	134	273.2	172	341.6	210	410.0
21	69.8	59	138.2	97	206.6	135	275.0	173	343.4	220	428.0
22	71.6	60	140.0	98	208.4	136	276.8	174	345.2	230	446.0
23	73.4	61	141.8	99	210.2	137	278.6	175	347.0	240	464.0
24	75.2	62	143.6	100	212.0	138	280.4	176	348.8	250	482.0
25	77.0	63	145.4	101	213.8	139	282.2	177	350.6	260	500.0
26	78.8	64	147.2	102	215.6	140	284.0	178	352.4	270	518.0
27	80.6	65	149.0	103	217.4	141	285.8	179	354.2	280	536.0
28	82.4	66	150.8	104	219.2	142	287.6	180	356.0	290	554.0
29	84.2	67	152.6	105	221.0	143	289.4	181	357.8	300	572.0
30	86.0	68	154.4	106	222.8	144	291.2	182	359.6	350	662.0
31	87.8	69	156.2	107	224.6	145	293.0	183	361.4	400	752.0
32	89.6	70	158.0	108	226.4	146	294.8	184	363.2	450	842.0
33	91.4	71	159.8	109	228.2	147	296.6	185	365.0	500	932.0
34	93.2	72	161.6	110	230.0	148	298.4	186	366.8	550	1,022.0
35	95.0	73	163.4	111	231.8	149	300.2	187	368.6	600	1,112.0
36	96.8	74	165.2	112	233.6	150	302.0	188	370.4	650	1,202.0
37	98.6	75	167.0	113	235.4	151	303.8	189	372.2	700	1,292.0

FORMS FOR REPORTING TESTS.

FILE CARDS.

Form 93.—Rev.

UNITED STATES DEPARTMENT OF AGRICULTURE,
OFFICE OF PUBLIC ROADS AND RURAL ENGINEERING,
Washington, D. C.

Sample No. .....

Report on .....
Identification mark .....
Known as .....
Submitted by .....
Examined for .....

		% by vol.	% by wt.	Character.
General character	Distillation—			
Specific gravity 25°/25°C	Water			
Melting point °C	First light oils...(–110°C)..			
Float test at ..... °C.....; at..... °C.....	Second light oils.(110–170°C)..			
Viscosity Engler at ..... °C..... c. c. specific...	Heavy oils.....(170–270°C)..			
Bitumen soluble in CS_2	Heavy oils.....(270–300°C)..			
Free carbon (insoluble in CS_2)	Pitch residue			
Ash				
.....	Total			

.....
.....
Remarks .....
.....
.....
Date received ....., Date reported .....

Analyst .....

Form 77.—Rev.

UNITED STATES DEPARTMENT OF AGRICULTURE,
OFFICE OF PUBLIC ROADS AND RURAL ENGINEERING,
Washington, D. C.

Sample No. .....

Report on .....
Identification mark .....
Known as .....
Submitted by .....
Examined for .....

General character	Loss at.....; °C.....hours .....
Specific gravity 25°/25°C	Character of residue .....
Melting point °C	Consistency of residue {Float test at..... °C..... {Penetration at 25 °C. (mm.)
Flash point °C	Bitumen soluble in CS_2 .....
Burning point °C	Organic matter insoluble .....
Viscosity at..... °C.....; c. c. specific.....	Inorganic matter insoluble .....
Ductility at 25°C. (cm.)	% of total bitumen insol. in 86° naphtha .....
Float test at.... °C. time....; at.... °C. time....	% of total bitumen insol. in CCl_4 .....
Penetration at 25°C. (mm.)	% fixed carbon .....
.....	

Remarks .....
.....
Date received ....., Date reported .....

Analyst .....

Form 67.—Rev.

UNITED STATES DEPARTMENT OF AGRICULTURE,
OFFICE OF PUBLIC ROADS AND RURAL ENGINEERING,
Washington, D. C.

Sample No. .....

Report on .....
Identification mark .....
Known as .....
Submitted by .....
Examined for .....
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Examined for—Continued.

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Remarks.....

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Date received....., Date reported.....

Analyst.....

REPORT BLANKS.

Form 104-B.

UNITED STATES DEPARTMENT OF AGRICULTURE,
OFFICE OF PUBLIC ROADS AND RURAL ENGINEERING,
Washington, D. C.

Date.....

Sample No. .....

Report on.....

Identification mark.....

Known as.....

Submitted by.....

Examined for.....

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Remarks.....

Form 109-B.

UNITED STATES DEPARTMENT OF AGRICULTURE,
OFFICE OF PUBLIC ROADS AND RURAL ENGINEERING,
Washington, D. C.

Date.....

Sample No. .....

Report on.....

Identification mark.....

Known as.....

Submitted by.....

Examined for.....

General characteristics.....

Specific gravity 25°/25°C.....

Melting point °C.....

Flash point °C.....

Burning point °C.....

Viscosity Engler at.....°C.....c. c. specific.....

Consistency { Float test.....°C., time.....; .....°C., time.....
Penetration 25 °C., 100 gms., 5 seconds.....

Loss at 163°C., 5 hours .....

Character of residue.....

Consistency of residue { Float test.....°C. time.....; .....°C., time.....
Penetration 25 °C., 100 gms., 5 seconds.....

Bitumen soluble in CS_2 (total bitumen).....

Organic matter insoluble.....

Inorganic matter insoluble.....

Per cent of total bitumen insoluble in 86° B. naphtha.....

Per cent of total bitumen insoluble in CCl_4.....

Per cent fixed carbon.....

Distillation—

	Character.	% by vol.	% by wt.
Water.....	.....	.....	.....
First light oils ..(–110°C.).....	.....	.....	.....
Second light oils (110°–170°C.).....	.....	.....	.....
Heavy oils.....(170°–270°C.).....	.....	.....	.....
Heavy oils.....(270°–300°C.).....	.....	.....	.....
Pitch.....	.....	.....	.....

Remarks.....